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RESEARCH ON METALLURGICAL CHARACTERISTICS AND PERFORMANCE OF MATERIALS USED FOR SLIDING ELECTRICAL CONTACTS

by W. H. Abbott and E. S. Bartlett

Prepared by
BATTELLE MEMORIAL INSTITUTE
Columbus, Ohio
for Electronics Research Center

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RESEARCH ON METALLURGICAL CHARACTERISTICS AND PERFORMANCE OF MATERIALS USED FOR SLIDING ELECTRICAL CONTACTS

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SUMMARY

This report summarizes the results of research conducted at Battelle during the period of May 1, 1968, to April 30, 1969. The primary objective of the program was to define the metallurgical or other materials characteristics which will be required for advanced, high reliability, low energy, sliding contact systems. These systems may operate in earth and space environments and at temperatures as high as 200°C.

Studies of the friction, wear, and contact resistance stability of nine wrought gold alloys and three electroplated gold systems were made in both N_2-20 O_2 and high vacuum (10^{-7}-10^{-8} torr). Sliding durations were 100 cycles in N_2-20 O_2 and 500 cycles in vacuum.

Environmental tests were made on each materials system. These consisted of (1) exposure to flowers of sulfur at 30°C, and (2) 1000-hour storage in N_2-20 O_2 at 200°C. Wear test and control specimens exposed in this manner were analyzed by contact resistance measurements and analysis of surface films.

All of the wrought gold alloys showed high friction (μ_k = 1.2-2.0) and wear rates in both air and vacuum. No correlation was apparent between wear rates, wear mechanism, and mechanical properties. Differences in wear and friction were attributed almost entirely to differences in surface chemistry (residual films and/or wear induced films) or surface energy of the alloy.

Contact resistance stability was shown to be closely related to wear mechanism and resistivity. High resistivity and fine wear particles always produced static contact resistance instability and high noise levels.

Several series of Au-Co electroplates showed much lower friction and wear and low noise levels compared to the wrought alloys. The improved performance of these materials was attributed to a unique surface chemistry consisting of an organic "lubricant" film codeposited with the electroplate.

All of the materials systems maintained low and stable contact resistance in a sulfur-bearing environment. Elevated temperature (200°C) exposure in air was identified as the major limitation of advanced materials systems. Of the wrought alloys tested only Au-19.0Ag, Au-0.5Zr, Au-2.5Zr, and Au-19.4Ag-1.64Zr

compositions maintained low contact resistance and no visible surface films after a 200°C exposure.

Substrate diffusion by a grain boundary or dislocation transport mechanism at 200°C limited the life of the Au-Co electroplates over both copper and nickel substrates. Rhodium was tentatively identified as a satisfactory diffusion barrier.

INTRODUCTION

The primary objective of this research program was to define the metallurgical or other materials characteristics which will be required for advanced, high reliability, low energy sliding contact systems. It was planned that the research would be directed toward contact systems designed for a relatively short sliding life (500 cycles maximum) in either earth or space environments. Such systems might, however, be required to exhibit long shelf-lives in an earth or space environment, possibly at temperatures as high as 200°C.

The results of these studies were expected to be directly applicable to the design of advanced electronic connectors. Therefore, primary emphasis was placed on the dry sliding characteristics of gold-base alloys operating as similar metal sliding contacts. Limited studies were also made on several dissimilar metal systems in order to define basic differences between similar and dissimilar metal sliding contact.

Gold electrodeposits have long been used in connector design. Limited selection of alloys and quality control problems attendant with the use of electroplates has prompted NASA to determine if wrought gold alloys offer any unique technical advantage that could lead to their wider use in advanced component design. For this reason, initial studies were made on the sliding characteristics of selected, wrought, gold-base alloys. The selection was based on earlier research at Battelle which indicated alloy systems that might meet the program objectives.

For comparison with the wrought alloys studies were planned on two types of electroplates. The Au-Co alloys were selected on the basis of an extensive history of use in connector systems. In spite of their wide use the reasons for their performance characteristics have not been defined nor has their value for high temperature service been assayed.

During elevated temperature service or storage, substrate diffusion through thin electroplates or clad wrought alloy systems was recognized as a potential reliability problem. Therefore, environmental studies of electroplates were designed to evaluate the effects of 200°C diffusion on contact resistance and surface film formation.

EXPERIMENTAL RESEARCH WORK

Materials Systems

Nine basic wrought alloy systems were selected for evaluation as similar metal sliding contacts. These include Au-19.0Ag*, Au-19.3Ag-2.05In, Au-12.1Cu, Au-12.3Cu-2.23In, Au-1.0Co, Au-1.0Co-1.0In, Au-0.5Zr, Au-2.5Zr, and Au-19.4Ag-1.64Zr. Silver was selected as an alloying addition to improve wear, but which would not degrade the elevated temperature oxidation resistance of gold. In spite of the fact that silver is one of the least effective strengthening additions to gold significant increases in hardness can be effected by ternary additions to Au-Ag alloys. Although silver additions to gold tend to give perceptible tarnish rates, up to about 30 at.% Ag the binary alloys can be considered as nontarnishing. The particular alloy composition of 19.0 wt.% Ag (30 at.% Ag) represents a good balance between improved wear and reasonably low tarnish rates.

Indium was selected as a ternary hardening addition mainly from considerations of surface film. Studies at Battelle have indicated that indium is very effective in decreasing the tarnish rates of Au-Ag alloys without seriously affecting oxidation resistance.

Copper was selected as a more effective solid solution hardening addition than silver. Although prior studies at Battelle had indicated that use of the Au-12.1 wt.% (30 at.%) Cu alloy would not be limited by tarnish reactions, its oxidation resistance at 200°C had not been evaluated.

Cobalt and zirconium were selected both as solid solution hardening additions and to provide alloy systems which might provide considerable precipitation hardening. The equilbrium precipitate in Au-Co alloys is known to be almost pure cobalt. In Au-Zr alloys the second phase is the compound Au_3Zr. These differences might be expected to have a pronounced effect on elevated temperature oxidation due differences in activity of the oxidizable constituent. Verification of this thesis eventually led to the addition of zirconium to the binary Au-Ag alloy.

In addition to the compositions listed above two complex wrought alloys were evaluated in the dissimilar metal sliding systems. These compositions were Au-14.0Cu-9.0Pt-5.0Ag-1.0Zn and Pd-13.8Cu-30.0Ag-10.0Au-10.0Pt-1.0Zn. These alloys are presently in wide use as brush materials in slip-ring systems due to their exceptionally good spring properties.

Two series of gold alloy electroplates were evaluated as similar metal sliding contacts. These had the nominal compositions Au-0.1Co and Au-1.0Co. Both alloys were plated over copper and nickel substrates to nominal thicknesses of 50, 100, and 200 microinches. These systems were selected on the

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* Compositions given in weight percentages.

basis of (1) wide use of the Au-Co alloy electroplates as contact materials, (2) possible differences between wrought and electroplated compositions which could provide valuable information on important material properties, and (3) possible effects of substrate mechanical properties.

Alloy preparation.--The wrought alloys were prepared as 50-60 gram charges by high-frequency induction melting in 13 mm ID x 19mm OD quartz tubing. Prior to melting, the tubes were evacuated, backfilled with ~ 250 mm Hg high-purity argon, and sealed. Each of the quartz tubes was in turn sealed in a 25 mm ID quartz tube under ~ 250 mm Hg argon. The purpose of the outer tube was simply to preclude the loss and/or contamination of the precious metal-alloy in the event of fracture of the container tube during melting.

Each alloy was heated to about 300°C above its liquidus temperature, held at this temperature for about 5 minutes with vigorous agitation, and then allowed to cool through the solidus temperature with continuous and vigorous agitation.

Similar fabrication procedures were employed to obtain 0.114-inch-diameter rod for all materials except the two complex brush materials which were considerably more difficult to fabricate. Except for these two alloys, the materials were first cold swaged to 0.400 inch diameter (39% RA*). At this point each alloy was given an homogenization anneal for 16 hours in flowing N_2 at 760 mm Hg followed by a water quench. The annealing temperatures were 700°C for the Au-Ag, and Au-Cu binary and ternary alloys and 800 C for the Au-Co, Au-Co-In, Au-Zr, and Au-Ag-Zr alloys.

Next, each alloy was cold swaged to 0.250 inch diameter (61% RA) and annealed for 1 hour in N_2 at the same temperature as used for the previous anneal. This was followed by cold swaging to 0.171 inch diameter and wire drawing to 0.114 inch diameter for a total final reduction of 79% RA. The Au-Ag-Zr alloy only was given an intermediate anneal at 0.171 of 800°C, 1 hour.

No lubricant was used during swaging but a lubricant was used for all wire drawing operations. This consisted of a water-soluble grade (Hangsterfers Laboratories, HE-2) mixed at a ratio of 7 parts lubricant to 1 part water.

Both of the complex brush materials were very difficult to fabricate from the cast condition. Therefore, a technique was employed which has frequently been used to fabricate materials prone towards failure under tensile stresses. This consisted of first placing each alloy in a mild steel swaging container. The dimensions of the container were 0.515 inch ID x 0.765 inch OD x 6.0 inches length x 5.5 inches cavity depth. The encapsulated specimens were cold swaged to container diameter of 0.500 inch OD which corresponded to a specimen diameter of 0.375-0.380 inch. At this point the steel was removed in 50 percent HNO_3 at room temperature. This was followed by a 10-minute etch of each specimen in aqua regia at room temperature.

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* % RA = percentage reduction by area.

Both of the brush alloys appeared to be of good quality after encapsulated swaging to ~ 0.375 inch (46% RA). Following an homogenization anneal for 16 hours at 850°C in N_2 the alloys were cold swaged to 0.302 inch diameter (35% RA). The alloys were annealed 1 hour at 850°C, cold swaged to 0.260 inch, annealed 1 hour at 850°C, cold swaged to 0.220 inch, annealed 1 hour at 850°C, and cold swaged to 0.121 inch at which point slight surface cracking was observed. It was decided to evaluate the materials in this condition corresponding to 56-60% RA in preference to annealing and cold working only ~ 10% to 0.114 inch diameter.

Specimens were prepared from each alloy as 1.5-inch lengths of 0.108-inch diameter rods. This was accomplished by centerless grinding which resulted in a surface finish of ~ 30-35 microinches CLA. Prior to testing, the specimens were finished and cleaned by a special metallographic technique developed at Battelle to insure minimum residual surface contamination. For example, detailed surface studies on pure gold prepared in this manner have shown only a residual film of about one monolayer of adsorbed water. The final surface roughness of all specimens was 5-10 microinches CLA.

Electroplates.--Several series of Au-0.1Co* and Au-1.0Co** electroplates were prepared. The substrate materials were cold worked OFHC copper and cold worked "A" nickel in the form of 0.105 to 0.108-inch-diameter x 1.5-inch-long rods. Nominal plating thicknesses were 50, 100, and 200 microinches.

Previous studies have established the fact that smooth substrates are highly desirable to obtain (1) minimum porosity and (2) maximum durability of thin electrodeposited coatings. Chemical polishing was used to obtain surfaces having initial roughness values of 5-7 microinches CLA for copper and 8-10 micro-inches for nickel. These values represent about the minimum roughness levels that could be attained for surfaces which were initially ground to approximately 20-25 microinches CLA. Studies showed that for the solutions and conditions listed in Table I, the optimum polishing time was about 240 seconds. Longer polishing times resulted in an increase in roughness as a result of etching.

A standard technique was established for cleaning and finishing of both copper and nickel as follows:

(1) Solvent clean racked specimens in boiling benzene.

(2) Chemical polish in 1-liter solution, 240 seconds.

(3) Rinse in three 1-liter portions of distilled water at 25°C.

(4) Dip in 1-liter 6N HCl for 60 seconds at 25°C.

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* Sel-Rex Autronex CI

** Sel-Rex Autronex C

TABLE I

CHEMICAL POLISHING SOLUTIONS

Reagent	Composition, volume percent	
	For Copper Polishing	For Nickel Polishing
Conc. HNO_3 (70%)	15.0	19.5
Conc. CH_3COOH (99%)	42.5	38.5
Conc. H_3PO_4 (85%)	42.5	38.5
Conc. H_2SO_4 (96%)	--	3.0
Conc. HCl (37%)	--	0.2
H_2O	--	0.3
Temperature, °C	70 ± 1	82 ± 1

(5) Rinse in 1-liter distilled water at 25°C.

(6) Dip in 1-liter 3.6N H_2SO_4 for 60 seconds at 25°C.

(7) Rinse in 1-liter distilled water at 25°C.

(8) Gold strike* 5 seconds at 10 amp/ft^2, 140 F.

(9) Rinse in 1-liter distilled water at 25°C.

(10) Dip in 1-liter 1.2N H_2SO_4 for 60 seconds at 25°C.

(11) Rinse in 1-liter distilled water at 25°C.

(12) Gold electroplate.

(13) Rinse in 1-liter distilled water at 25°C, then in 1-liter distilled water at 80°C.

(14) Rinse in 1-liter ethanol at 60°C.

(15) Air dry and store in glass vials.

The conditions of operation for each electroplating solution are given in Table II. Solutions were contained in a glass and Tygon tubing system which was continuously filtered to a particle size of 1-3 microns. Vigorous agitation of the baths was obtained from the pump effluent. Pure platinum was used as the anode material in sufficient amount to give an anode:cathode area ratio of about 3:1.

TABLE II

OPERATING CONDITIONS OF GOLD ELECTROPLATING SOLUTIONS

Solution	Temp., °C	pH	Specific Gravity °Be'	Current Density, amp/ft^2	Bath Composition g/ℓ	
					Au	Co
Aurobond TN	60	3.70	12.	10.	1.73	--
Autronex CI	34	4.05	10.	10.	8.66	0.10
Autronex C	34	3.55	10.	10.	9.20	0.50

* Sel-Rex Aurobond TN.

Specimens were rack mounted and plated in groups of nine. The 304 stainless steel rack was coated with polyvinyl chloride. Each specimen was held horizontally by cone ended, cantilever spring arms which pressed against the end faces of each rod.

Plating thicknesses for the Autronex CI deposits were initially calculated on the basis of a deposition rate of 10 microinches per minute as indicated by the supplier for the plating conditions of Table II. Subsequent studies revealed that the actual cathode efficiency, i.e., plating rate, was considerably greater than expected. The experimental value was 58.7 mg/amp-min which for a current density of 10 amp/ft^2 corresponds to 12.9 microinches per minute.

Plating rates were also found to be higher than expected for the Autronex C solutions. Experimental values were 39.3 mg/amp-min and 8.65 microinches per minute at 10 amp/ft^2.

Chemical analysis.--Spectrographic analyses for impurities in both the wrought alloys and the electroplates are given in Tables III and IV. Major impurities in most of the wrought alloys were silver, iron, and silicon. The source of silver was the pure gold melting stock which contained about 0.01 percent ailver. Late in the program a higher purity (99.99) gold was used to prepare alloys Au-8, Au-11, and Au-12. This accounts for the lower silver contents in alloys Au-8 and Au-11. Iron is an impurity commonly encountered in fabricated alloys and was probably introduced as die pickup during swaging. Silicon was found only in the zirconium-containing alloys. The reduction of small amounts of SiO_2 from the melting capsules by zirconium during melting was the most probable cause of silicon contamination.

Silver, copper, and titanium were the major impurities in the Au-Co electroplates. The data for silver and titanium are resonably accurate but the values for copper may be high. This may be due to incomplete stripping of the copper substrate from the electroplate prior to the analysis.

Major alloying constituents were analyzed by wet chemical techniques with the results given in Table V. The compositions of all alloys except Au-7 were very close to the intended values. The gold-base alloy--Au-7--was apparently 1.9 percent low in copper, 1.0 percent low in gold, and 3.3 percent high in platinum. No explanation is available for these relatively large variations in composition. One contribution was almost certainly the analytical technique as considerable difficulty was encountered in effecting complete separation of the elements in this alloy. On the basis of resistivity data it is believed that the actual composition of this alloy was close to nominal.

Cobalt analysis of the electroplates gave values considerably different than the nominal values of 0.1 and 1.0 weight percent. Experimental values were 0.18-0.2 percent cobalt for Autronex CI (0.1 nominal) and 0.76 percent cobalt for Autronex C (1.0 nominal). These results were not too surprising based on earlier studies of acid Au-Co processes at Battelle. Cobalt contents have been shown to depend on both current density and local flow conditions around the specimens. The Autronex CI process was found to be much more

TABLE III

EMISSION SPECTROGRAPHIC ANALYSIS OF WROUGHT ALLOYS

Designation	Material At. %	Material Wt. %	Wt. % of Specified Element Au	Ag	In	Cu	Si	Mn	Fe	Mg	Pb	Ni	Al	Cd	Ti	Pd	Ca	Cr	Sn
Gold Powder	--	--	--	.01	<.001	.001	<.001	<.001	.001	<.001	<.001	<.001	<.001	<.003	.003	<.001	<.001	<.001	<.001
Au-1A	Au-30Ag	Au-19.0Ag	--	--	<.001	.005	.001	<.001	.01	<.001	<.001	<.001	.001	.001	.002	<.001	<.001	<.001	<.001
Au-1C	Au-30Ag	Au-19.0Ag	--	--	<.001	.005	<.001	<.001	.03	<.001	<.001	<.001	<.001	.001	<.001	<.001	<.001	<.001	<.001
Au-2A	Au-30Cu	Au-12.1Cu	--	.02	<.001	--	<.001	<.001	.02	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Au-2B	Au-30Cu	Au-12.1Cu	--	.02	<.001	--	<.001	<.001	.02	<.001	<.001	<.001	<.001	<.001	.002	<.001	<.001	<.001	<.001
Au-2C	Au-30Cu	Au-12.1Cu	--	.02	<.001	--	<.001	<.001	.01	<.001	<.001	<.001	<.001	<.001	.002	<.001	<.001	<.001	<.001
Au-3	Au-30Ag-3In	Au-19.3Ag-2.05In	--	--	--	.003	.001	<.001	.03	<.001	.002	<.001	.003	.001	.002	<.001	<.001	<.001	<.001
Au-4	Au-30Cu-3In	Au-12.3Cu-2.23In	--	.02	--	--	<.001	<.001	.01	<.001	.002	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Au-5	Au-3.27Co	Au-1.0Co	--	.02	<.001	.002	<.001	<.001	.01	<.001	<.001	.003	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Au-6	Au-3.25Co-1.66In	Au-1.0Co-1.0In	--	.02	--	.002	<.001	<.001	.01	<.001	<.001	.003	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Au-7	Au-32Cu-6.5Ag-6.5Pt-2.0Zn	Au-14Cu 9.0Pt-5.0Ag-1.0Zn	--	--	<.001	--	<.001	<.001	.02	.001	<.001	<.001	<.001	<.001	<.001	.002	<.001	<.001	<.001
Au-8	Au-1.07Zr	Au-0.5Zr	--	.005	<.001	.002	.01	<.001	.003	<.001	<.001	<.001	<.001	<.001	.001	.003	<.001	<.001	<.001
Au-11	Au-5.26Zr	Au-2.5Zr	--	.005	<.001	.001	.03	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Au-12	Au-30Ag-3Zr	Au-19.4Ag-1.64Zr	--	--	<.001	.002	.02	<.001	.002	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Pd-1	Pd-29Ag-23Cu 5.5Au-5.5Pt-2.0Zn	Pd-13.8Cu 30Ag-10Au-10Pt-1.2Zn	--	--	<.001	--	.003	<.001	.02	<.001	<.001	<.001	<.001	<.001	.003	--	<.001	<.001	<.001

TABLE IV

EMISSION SPECTROGRAPHIC ANALYSIS OF Au-Co ELECTROPLATES

Nominal Alloy Composition, wt.%	Wt.% of Specified Element[a]										
	Ag	Cu	Si	Mn	Fe	Mg	Pb	Ni	Al	Ti	Cr
Au-0.1Co[b]	<0.001	0.05	<0.001	<0.001	<0.001	<0.001	0.002	<0.001	<0.001	0.005	<0.001
	<0.001	0.03	<0.001	<0.001	<0.001	<0.001	0.002	<0.001	<0.001	0.005	<0.001
	<0.001	0.03	<0.001	<0.001	0.002	<0.001	0.003	<0.001	<0.001	0.005	<0.001
Au-1.0Co	0.01	0.03	0.003	0.001	0.001	<0.001	0.001	0.001	0.001	0.005	0.001
	0.01	0.02	0.003	0.001	0.001	<0.001	0.001	<0.001	0.002	0.005	<0.001

(a) Impurity analysis only.

(b) Sel-Rex Autronex CI.

(c) Sel-Rex Autronex C.

TABLE V

CHEMICAL ANALYSIS OF WROUGHT ALLOYS

Material		Analyzed Composition, wt.%								
Designation	Nominal Wt.%	Au	Ag	Cu	Co	In	Pd	Pt	Zr	Zn
Au-1A	Au-19.0Ag	80.9	19.1	--	--	--	--	--	--	--
Au-1C	Au-19.0Ag	81.0	19.0	--	--	--	--	--	--	--
Au-2A	Au-12.1Cu	88.0	--	12.0	--	--	--	--	--	--
Au-2B	Au-12.1Cu	88.1	--	12.0	--	--	--	--	--	--
Au-2C	Au-12.1Cu	87.9	--	12.2	--	--	--	--	--	--
Au-3	Au-19.3Ag-2.05In	78.1	19.5	--	--	1.80	--	--	--	--
Au-4	Au-12.3Cu-2.23In	85.5	--	12.1	--	2.27	--	--	--	--
Au-5	Au-1.0Co	98.8	--	--	0.91	--	--	--	--	--
Au-6	Au-1.0Co-1.0In	98.0	--	--	0.91	1.05	--	--	--	--
Au-7	Au-14Cu-9Pt-5Ag-1Zn	70.0	5.04	12.1	--	--	--	12.3	--	0.88
Au-8	Au-0.5Zr	99.5	--	--	--	--	--	--	0.51	--
Au-11	Au-2.5Zr	97.8	--	--	--	--	--	--	2.21	--
Au-12	Au-19.4Ag-1.64Zr	79.6	19.0	--	--	--	--	--	1.40	--
Pd-1	Pd-13.8Cu-30Ag-10Au-10Pt-1.2Zn	10.2	29.4	13.7	--	--	35.3	10.4	--	0.98

TABLE VI

RESISTIVITY OF WROUGHT PRECIOUS METAL ALLOYS

Material		Resistivity Microhm-cm @ 25° C		
Designation	Nominal Composition, wt.%	#1	#2	Number of Phases
Au-1A	Au-19.0Ag	9.450	9.410	1
Au-1C	Ditto	9.348	9.259	1
Au-2A	Au-12.1Cu	12.328	12.565	1
Au-2B	Ditto	12.062	12.002	1
Au-2C	"	11.894	11.971	1
Au-3	Au-19.3Ag-2.05In	13.193	13.362	1
Au-4	Au-12.3Cu-2.23In	15.787	15.756	1
Au-5	Au-1.0Co	19.215	18.979	1
Au-6	Au-1.0Co-1.0In	21.053	21.118	>1
Au-7	Au-14Cu-9Pt-5Ag-1Zn	23,727	22.493	1
Au-8	Au-0.5Zr	14.659	--	1
Au-11	Au-2.5Zr	44.620	--	2
Au-12	Au-19.4Ag-1.64Zr	16.999	--	>1
Pd-1	Pd-13.8Cu-30Ag-10Au-10Pt-1.2Zn	32.719	32.827	1

sensitive to these variations than the Autronex C. Increases in current density and/or decreases in flow rates tended to increase the cobalt content.

By close control of the plating conditions it was possible to produce plates of reproducible composition and properties. Although it was possible to produce an Au-0.1Co composition, the conditions necessary to obtain this cobalt content were not consistent with a high quality electroplate. Low current densities (< 7 amp/ft^2) and/or high flow rates gave dull, porous, deposits characterized by an anomalous, nodular growth as shown in Figure 1. A surface typical of the specimens being tested is shown in Figure 2.

Resistivity.--Experimental resistivity values for the wrought alloys are given in Table VI. No unexpected effects of alloying were observed.

Results for the binary Au-Ag, Au-Cu, and Au-Co alloys are in good agreement with the data of Wise (Ref. 1). Data were not readily available for the Au-Zr system but it is apparent that zirconium additions increase the resistivity of gold at a greater rate than many other elements. For small additions the rate of increase was estimated to be 11.4$\mu\Omega$-cm/at.% compared to $\sim$5.7$\mu\Omega$-cm/at.% for Au-Co, and 9$\mu\Omega$-cm/at.% for Au-Fe (Ref. 1).

On the basis of these data the alloy Au-11 (Au-2.5 wt.% Zr = Au-5.3 at.% Zr) should have shown a resistivity of about 63$\mu\Omega$-cm instead of 44.6$\mu\Omega$-cm. This low value was explained by microstructural examination which showed that the alloy was not in the solid solution condition. It was discovered that the Au-2.5Zr alloy had been process annealed at 800°C instead of the intended 900°C. The lower temperature is in the two-phase field for this alloy meaning that some precipitation of Au_3Zr occurred during annealing. At 800°C the maximum solubility of zirconium in gold is 2.1 weight percent (4.4 at.%). Assuming that this amount was retained in solution the expected resistivity of $\sim$ 48$\mu\Omega$-cm is in reasonably good agreement with the experimental value.

Studies were not made of the effects of heat treatment and precipitation on resistivity. However, during the research each alloy was subjected to a 1000-hour exposure at 200°C in N_2-20 O_2. This provided an opportunity to at least evaluate the stability of each alloy system under extreme exposure conditions.

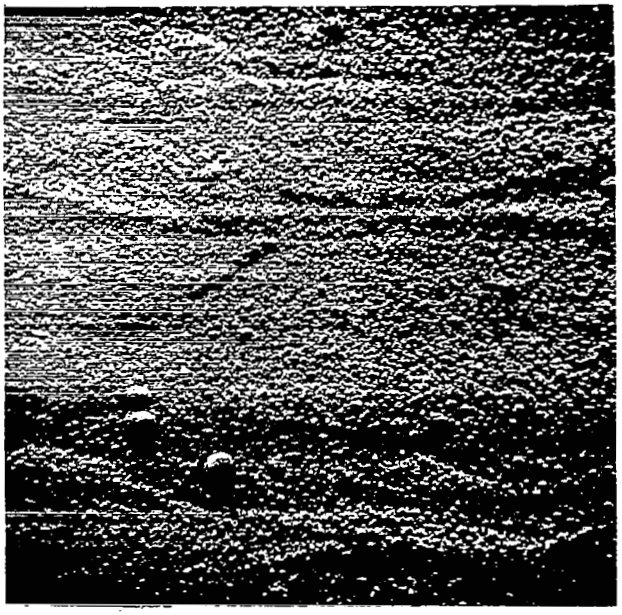
The results of these measurements are given in Table VII. Precipitation was considered thermodynamically favorable only in all the cobalt and zirconium-containing alloys. That precipitation did occur at 200°C is shown in the large resistivity changes for alloy Au-5 and Au-6. It is estimated that in 1000 hours at 200°C about 65 percent of the cobalt was precipitated from solution in alloy Au-5 leaving a matrix composition of $\sim$ Au-0.35 wt.% Co. Indium additions to Au-Co apparently increased the rate of precipitation.

The zirconium-containing alloys showed much better stability at 200°C. The small apparent decrease in alloy Au-8 could be attributed to either a very small amount of precipitation or an "annealing-out" of lattice defects. The latter is believed to account for the small resistivity decreases in alloys Au-1, 2, 3, and 4.

TABLE VII

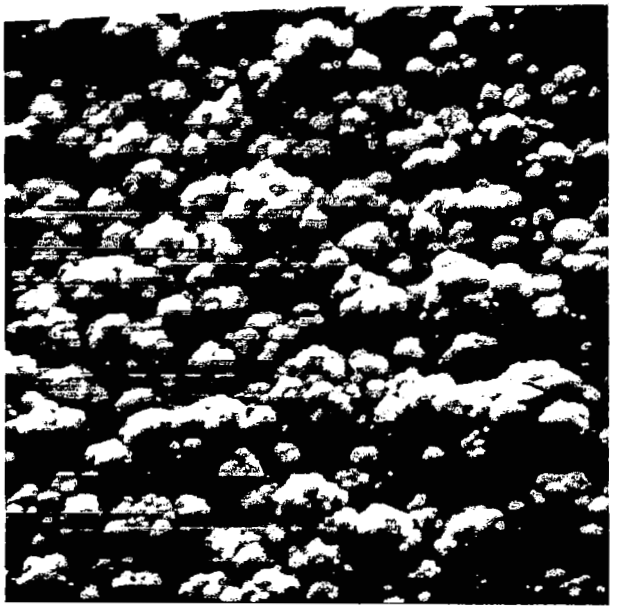
RESISTIVITY OF 1000 HR, 200°C OXIDATION SPECIMENS

Material		Resistivity, μΩ-cm @ 25°C	
Designation	Nominal Composition, wt.%	Initial	After Oxidation
Au-1A	Au-19.0Ag	9.450	9.179
		9.410	9.137
Au-2B	Au-12.1Cu	12.328	11.777
		12.565	11.775
Au-3	Au-19.3Ag-2.05In	13.362	12.524
		13.193	12.412
Au-4	Au-12.3Cu-2.23In	15.787	15.731
		15.756	15.728
Au-5	Au-1.0Co	19.215	10.159
		18.979	9.993
Au-6	Au-1.0Co-1.0In	21.053	7.255
		21.118	7.415
Au-8	Au-0.5Zr	14.659	13.754
Au-11	Au-2.5Zr	44.620	38.627
Au-12	Au-19.4Ag-1.64Zr	16.999	16.447



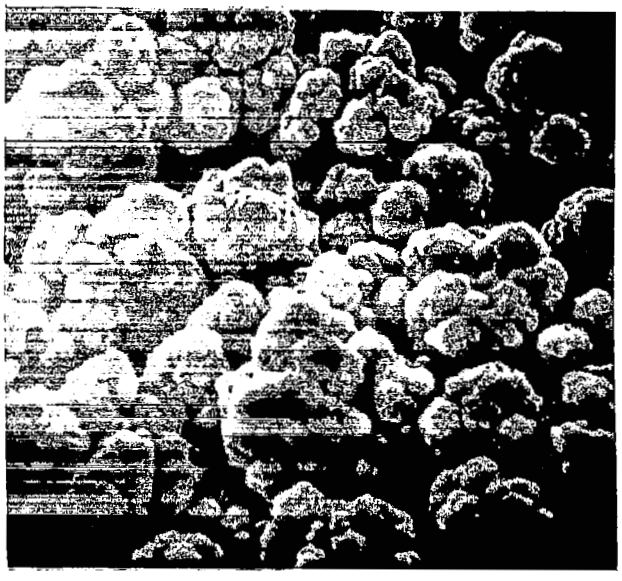
500X

a. 10 Amp/Ft^2, High Agitation



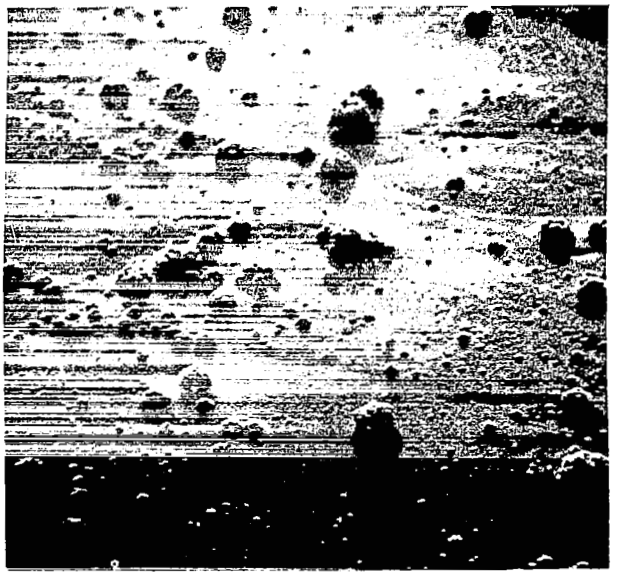
5000X

b. 10 Amp/Ft^2, High Agitation



10, 000X

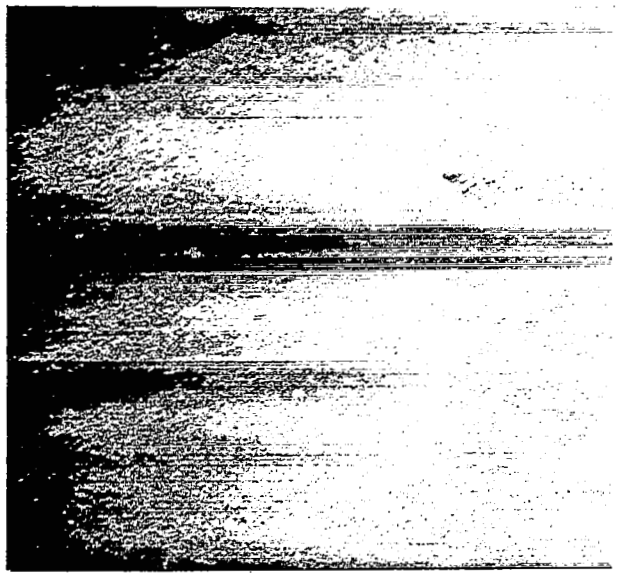
c. 10 Amp/Ft^2, High Agitation



1000X

d. 20 Amp/Ft^2, High Agitation

FIGURE 1. NODULAR GROWTH OF 100-MICROINCH Au-0.1Co ELECTROPLATES

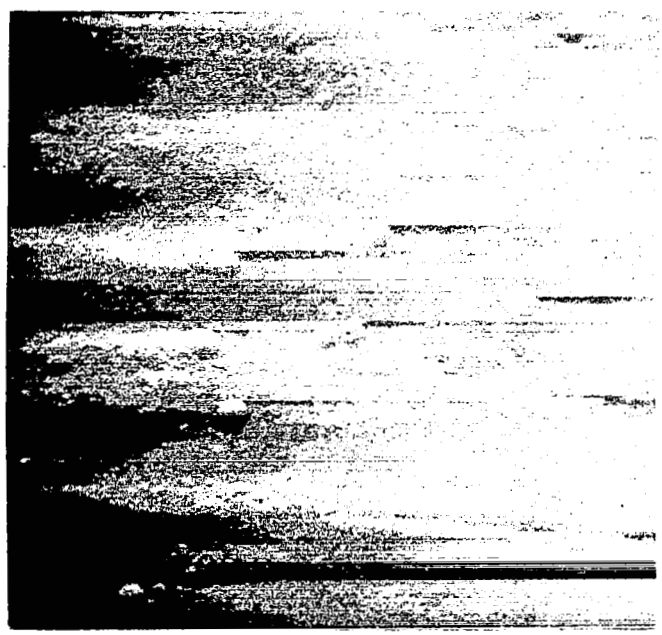


2000X

a. 200 Microinches

2000X

b. 100 Microinches



2000X

c. 50 Microinches

FIGURE 2. SCANNING ELECTRON MICROGRAPHS OF SURFACE OF Au-0.1Co ELECTROPLATES ON COPPER PRODUCED AT 10 AMP/FT^2 AND LOW AGITATION

The resistivity decrease in alloy Au-11 was too great to be due to reductions in lattice defects. A small amount of precipitation probably occurred at 200°C but it is evident that the precipitation rate must be very low.

These results may have particular significance for the development of contact materials for elevated temperature service. It is apparent that in applications requiring stable mechanical and electrical properties the Au-Co alloys may not be satisfactory. Even if Au-Co could be heat treated to high hardness levels, the data suggest that a drastic loss of hardness might result at 200°C as a result of averaging. The Au-Zr alloys do not appear to present this problem. It seems probable that if these alloys were heat treated to obtain desirable mechanical properties, they would be retained after an extended 200°C exposure. In addition to obtaining better mechanical properties by heat treatment, it should be possible to obtain much lower values of resistivity and hence lower and stable contact resistance.

Microhardness.--Microhardness values for all of the wrought alloys are given in Table VIII. On an atomic percentage basis cobalt and zirconium were much more effective hardeners for gold than silver or copper. Zirconium was the most effective hardener both as a binary addition to gold and as a ternary addition to Au-Ag.

Selected tests were made to determine the effects of heat treatment on microhardness with the results given in Table IX. The data were obtained to give an indication of the probable aging response of the alloys containing cobalt or zirconium. For the Au-Co and Au-Co-In alloys initial tests indicated that a temperature of 400°C might give nearly the best combination of reasonable kinetics and a small hardening increment. The data, however, show that at 400°C the hardening increment was very small. Maximum hardness was probably approached after 90 minutes at 400°C for Au-Co while the Au-Co-In alloy showed evidence of overaging within 60 minutes.

The Au-0.5Zr alloy showed no evidence of precipitation hardening at temperatures of 300-500°C. Hardening was apparently offset by recrystallization and/or overaging which was pronounced at temperatures of 400°C or greater.

Only the Au-2.5Zr alloy ($\sim$ 2.1Zr in solution) could be appreciably hardened above the cold worked level by heat treatment. The optimum heat treatment temperature is probably near 300°C, but neither the optimum time or the maximum hardening increment have been determined.

Several additional experiments were made in which the Au-2.5Zr alloy was first solution annealed at 950°C and heat treated from this condition. Tests were made only for exposures of 1 hour at 300, 400, 500, and 600°C. From an annealed hardness of H_K = 75, hardnesses after 1 hour were 92 at 300°C, 175 at 400°C, 211 at 500°C, and 188 at 600°C.

It is apparent from these data that significant hardening can be achieved at the Au-2.5Zr alloy from the annealed condition. As expected precipitation rates are lower for the annealed condition but it is probable that maximum

TABLE VIII

MICROHARDNESS OF WROUGHT ALLOYS

Material			
Designation	Nominal Composition, wt.%	Cold Work, %	Microhardness $(H_k)_{100}$
Au-1A	Au-19.0Ag	79	110
Au-1C	Ditto	79	112
Au-2A	Au-12.1Cu	79	235
Au-2B	Ditto	79	225
Au-2C	"	79	233
Au-3	Au-19.3Ag-2.05In	79	157
Au-4	Au-12.3Cu-2.23In	79	235
Au-5	Au-1.0Co	79	102
Au-6	Au-1.0Co-1.0In	79	131
Au-7	Au-14Cu-9Pt-5Ag-1Zn	56	375
Au-8	Au-0.5Zr	79	118
Au-11	Au-2.5Zr	79	180
Au-12	Au-19.4Ag-1.64Zr	56	194
Pd-1	Pd-13.8Cu-30Ag-10Au-10Pt-1.2Zn	60	330

TABLE IX

HEAT TREATMENT OF WROUGHT PRECIOUS METAL ALLOYS

Material			Microhardness $(H_k)_{100}$ After Heat Treatment[(a)] for Indicated Time, min.						
Designation	Nominal Composition, wt.%	Temp., °C	0	15	30	45	60	90	1440
Au-3	Au-19.3Ag-2.05In	700	157.	--	--	--	55.6	--	--
Au-4	Au-12.3Cu-2.23In	700	235.	--	--	--	144.	--	--
Au-5	Au-1.0Co	400	107.	112.	117.	117.	118.	119.	--
		800	107.	--	--	--	44.2	--	--
Au-6	Au-1.0Co-1.0In	400	131.	132.	130.	132.	128.	127.	--
		700	131.	--	--	--	48.4	--	--
Au-7	Au-14Cu-9Pt-5Ag-1Zn	850	375.	--	--	--	327.	--	--
Au-8	Au-0.5Zr	300	118.	--	103.	--	101.	--	107.
		400	118.	--	58.3	--	58.5	--	--
		500	118.	--	48.4	--	48.0	--	--
Au-11	Au-2.5Zr	300	179.	--	--	--	223.	--	--
		400	179.	--	119.	--	125.	--	--
		500	179.	--	108.	--	100.	--	--
		600	179.	--	107.	--	107.	--	--
		700	179.	--	108.	--	100.	--	--
Au-12	Au-19.4Ag-1.64Zr	300	194.	--	161.	--	165.	--	--
		400	194.	--	121.	--	115.	--	--
		500	194.	---	97.8	--	98.6	--	--
		600	194.	--	84.8	--	84.8	--	--
		950	194.	--	--	--	81.8	--	--
Pd-1	Pd-13.8Cu-30Ag-10Au-10Pt-1.2Zn	850	330.	--	--	--	204.	--	--

(a) Heat treated from cold worked condition.

hardness levels may be as great as those attained for the cold worked condition. For future studies of this alloy, heat treatment temperatures of 400-500°C are recommended.

Microhardness measurements on specimens exposed for 1000 hours at 200°C are given in Table X. The hardness increases in the Au-Cu and Au-Cu-In systems may be due to a low temperature ordering reaction. Slight hardness increases in the cobalt-containing alloys were attributed to precipitation of cobalt and/or indium from solution.

TABLE X

MICROHARDNESS OF 1000 HOUR, 200°C OXIDATION SPECIMENS

Material		Microhardness, $(H_K)_{100}$	
Designation	Nominal Composition, wt.%	Cold Worked[a]	200°C, 1000 hours
Au-1A	Au-19.0Ag	110.	94.7
Au-2B	Au-12.1Cu	225.	232.
Au-3	Au-19.3Ag-2.05In	157.	110.
Au-4	Au-12.3Cu-2.23In	235.	257.
Au-5	Au-1.0Co	107.	116.
Au-6	Au-1.0Co-1.0In	131.	134.
Au-8	Au-0.5Zr	118.	110.
Au-11	Au-2.5Zr	179.	195.
Au-12	Au-19.4Ag-1.64Zr	194.	145.

Based on resistivity, hardness data, and microstructural examination it is believed that no significant precipitation occurred at 200°C for Au-0.5Zr. The small hardness decrease may have been due to the onset of recrystallization. The Au-2.5Zr alloy definitely gave evidence of precipitation hardening at 200°C. This is in agreement with resistivity data.

Microhardness measurements on the Au-Co electroplates gave median values of $(H_K)_{10}$= 172 for Autronex CI and $(H_K)_{10}$ = 195 for Autronex C. The latter value is slightly lower than the range of 200-240 quoted by Sel-Rex. The actual difference is even greater since a correction factor of ~ 10 percent (Ref.2) downward should be applied to the present data in order to compare measurements at 10 grams with Sel-Rex data for 25 grams. Similar corrections should be made to the data for Autronex CI but the "corrected" value of H_K = 155 is within the expected range of 130-170.

Plating thickness measurements.--Plating thicknesses were measured from metallographic cross sections. Median values for the nominal 50, 100, and 200 microinch Autronix CI deposits were 81, 135, and 250 microinches respectively. As indicated earlier the higher values were due to higher than expected plating rates. The experimental values of plating rates determined gravimetrically

are in good agreement with the measured plating thicknesses.

Thicknesses for the Autronex C deposits were 55, 102, and 204 microinches. These values are in good agreement with measured plating rates which were determined before the test specimens were plated.

Porosity.--Porosity of the electrodeposits was determined by standard electrographic printing techniques. Kodak dye transfer paper was used with the following electrolyte:

1.0 wt.% NaCl
5.0 wt.% Na_2CO_3
Balance H_2O.

Prints were made at a current density of 10 ma/cm^2 at 6v d-c open-circuit potential and an electrolysis time of 30 seconds. Each print was developed in a solution of 1 wt.% dimethylglyoxime in ethyl alcohol. Pores were counted under a 40X stereo viewer.

Results for both series of Au-Co electroplates are given in Table XI together with surface roughness measurements.

TABLE XI

POROSITY MEASUREMENTS OF Au-Co ELECTROPLATES

Electroplate	Sub-strate	Roughness, microinches CLA	Plating Thickness, microinches	Pore Density, number per cm^2
Autronex CI	Cu	5.5	81	4,4,10,11,21
	Cu	5.5	135	0,2,3,4,7
	Cu	5.0	250	0,1,1,1,2
Autronex CI	Ni	12.	81	2,3,3,2,2
	Ni	12.	135	0,0,1,1,2,3,4,4,6,13
	Ni	12.	250	0,0,0,1,1,1,2,2,2
Autronex C	Cu	6.5	55	6,9,10,13,13,15,15,15,25
	Cu	6.2	102	0,0,1,3,4,5,6,6,11
	Cu	6.5	204	0,1,2,2,2,2,2
Autronex C	Ni	8.3	55	2,2,4,4,6,7,9,9
	Ni	7.5	102	0,0,0,2,3,3,4
	Ni	10.	204	0,0,0,0,0,0,0,1

Sliding Contact Studies

Test conditions.--All sliding tests were made under closely controlled environmental conditions of either high purity $N_2$20 O_2 or high vacuum. The N_2-20 O_2 was mixed from high-purity nitrogen and oxygen (Matheson) in an all-glass and stainless steel dilution system. Maximum impurity content of the gas was estimated to be < 10 ppm, with argon and CO_2 as the primary impurities. All tests in this environment were conducted under a continuous gas flow of ~7 ml/minute.

Vacuum environments were obtained from a 600 ℓ/sec Ultek ion pumping system. Vacuum sliding tests were made in a continuously pumped system. Neither the specimens nor the test chamber were baked prior to testing as it was intended that any unique effects of residual surface films should be reflected in the measurements.

The test chamber which was available from earlier studies at Battelle was designed according to ultrahigh vacuum principles. Stainless steel, glass (viewing ports), and alumina (insulators, electrical feed-throughs) were the only materials of construction. This assured virtually complete elimination of contaminants, particularly organics. Typical test vacuum was about 10^{-7} torr.

The contacts were mounted in a 90-degree crossed-rod configuration with one contact movable in reciprocating sliding over a 1 cm wear track. The "stationary" contact was dead-weight loaded to provide a constant and known normal contact force. This "stationary" assembly was mounted on a parallelogram arrangement of four leaf springs to which was attached a differential transformer (LVDT) core. Movement of the "stationary" contact by a horizontal frictional force gave a corresponding movement of the LVDT core. This in turn produced an electrical output from a matched concentric coil mounted outside the test chamber. The DC component of the coil output gave a very accurate measurement of friction forces.

Contact resistance was measured under dry circuit conditions by the four-wire technique. This provided a true measurement of constriction and film resistance without lead wire and/or bulk resistance contributions.

Both friction and contact resistance (noise) were continuously and simultaneously recorded on an optical oscillograph (Sanborn 4500) which was driven by a Sanborn 658-3400 medium gain amplifier. A Sanborn transducer amplifier (Model 311A) was used to obtain 2400 Hz excitation of the LVDT and to provide an amplified DC output to the recorder. A Hewlett-Packard 8875A high gain amplifier was used as a preamplifier for measuring contact noise.

A summary of the conditions of testing is given in Table XII. As shown, specimens were in sliding contact in two environments--N_2-20 O_2 at 760 mm Hg and high vacuum. Tests in N_2-20 O_2 were made for 100 cycles while vacuum tests were extended to 500 cycles. The 100 cycle and control specimens were subsequently given static exposures of (1) 1000 hours at 200°C in N_2-20 O_2 and (2) 120 hours in flowers of sulfur at 30°C. Separate specimens were used in each test. Specimen evaluation included contact resistance measurements and electrolytic reduction of surface films.

TABLE XII

TEST CONDITIONS FOR SLIDING CONTACT STUDIES

Variable	Range
Contact Force	1-25 grams for initial studies; constant force of 14 grams for detailed materials evaluation
Sliding Speed	0.01-1 cm/sec for initial studies; constant speed of 1 cm/sec for detailed materials evaluations
Environment	(1) N_2-20 O_2 at 760 mm Hg at 25°C for 100 cycles
	(2) N_2-20 O_2 at 760 mm Hg at 200°C for 1000 hours for both best and control specimens
	(3) N_2-20 O_2-40 ppb S_8 at 30°C for 100 hours for both test and control specimens
	(4) High vacuum (10^{-7} - 10^{-8} torr) at 25°C for 500 cycles
Electrical	10 mv dc open circuit voltage, 6 ma current

Initial studies were made on the alloys Au-1A and Au-2B to evaluate the effects of (1) contact force and (2) sliding speed over the range given in Table XII. As expected there were no effects on friction, wear, or static contact resistance stability. Noise levels showed a definite increase with increasing sliding speed and/or decreasing contact force. However, this was believed to be almost entirely a device effect rather than a basic change in wear mechanism. In consideration of these factors constant values of sliding speed and contact force were maintained in all subsequent tests at 1 ± 0.1 cm/sec and 14 ± 0.5 grams respectively.

Friction and contact resistance in N_2-20 O_2.--The results of all sliding tests conducted in the N_2-20 O_2 environment are summarized in Table XIII. Only the initial (Cycle No. 1) and final (Cycle No. 100) sliding parameters have been tabulated, as these are believed to be of the greatest practical importance. Initial friction values give a very sensitive indication of the surface chemistry of the materials. Final values give a measure of the effects of surface film disruption by the wear process as well as an increased ploughing contribution to friction.

During 100 cycles of sliding in N_2-20 O_2 and also 500 cycles in vacuum the friction slowly but continuously increased. The final values, therefore, represent the maximum values to be expected for sliding durations up to the number of test cycles studied.

Only the coefficients of kinetic friction, μ_k, are given in Table XIII. For connector-type applications the static coefficients are of equal or greater importance since these values determine maximum insertion and withdrawal forces. The coefficient of static friction which is a measure of microasperity adhesion and degree of junction growth is not a constant but may vary considerably during even a single wipe over a wear track. On the basis of these and other studies it has been found that for most gold alloys (> 60-70 wt.% gold) sliding under clean conditions the maximum value of μ_s is generally 40-50 percent greater than μ_k. This was found to be true for tests in both N_2-20 O_2 and vacuum.

A review of the data in Table XIII shows that as a group the wrought gold alloys gave similar and relatively high values of friction. With the exception of the Au-12.1Cu alloy (Tests 39 and 40) these alloys gave initial values of μ_k = 1.2-2.0. The Au-12.1Cu alloy gave only slightly lower values of $\mu_k \simeq 1.0$.

No systematic variation in friction with mechanical properties was apparent. Although the softest alloy (Au-1.0Co, H_K = 107) gave the highest friction, the hardest alloy (Au-Cu-In, H_K = 235) did not give the lowest friction. These results are in good agreement with earlier studies at Battelle on precious metal contact alloys. For materials having a much wider range of mechanical properties there is generally no correlation between friction and bulk mechanical properties.

The differences among friction values which were observed are believed to be due mainly to surface phenomena--specifically surface film characteristics.

TABLE XIII

SUMMARY OF SIMILAR-METAL SLIDING CONTACT TEST DATA FOR N_2-20 O_2 ENVIRONMENT

Material		Test	Coefficient of Friction, μ_k		Static Contact Resistance, mΩ				Sliding Contact Noise, Peak to Peak, mΩ	
Designation	Nominal Composition	No.	μ_1	μ_{100}	$(R_1)_{med.}$	ΔR	$(R_{100})_{med.}$	ΔR	ΔR_1	ΔR_{100}
Au-1A	Au-19.0Ag	37	1.60	2.22	6.3	5.6	3.7	1.9	5.8	4.7
Au-1A	Au-19.0Ag	38	1.74	2.48	6.4	4.9	3.1	1.1	4.9	4.7
Au-2B	Au-12.1Cu	39	1.02	1.44	6.8	5.4	8.8	2.2	7.0	7.0
Au-2B	Au-12.1Cu	40	0.98	1.96	7.8	6.8	5.6	4.3	9.3	7.0
Au-3	Au-19.3Ag-2.5In	41	1.25	1.50	10.4	8.8	7.4	3.0	6.9	6.9
Au-3	Au-19.3Ag-2.05In	42	1.24	1.74	9.2	8.3	7.0	4.6	8.3	5.9
Au-4	Au-12.3Cu-2.23In	43	1.14	1.77	13.2	24.7	10.4	4.5	18.9	9.4
Au-4	Au-12.3Cu-2.23In	44	1.47	1.59	12.4	20.0	10.9	4.3	8.1	9.3
Au-3	Au-19.3Ag-2.05In	45	1.38	1.57	8.0	8.6	6.7	3.0	5.8	5.7
Au-3	Au-19.3Ag-2.05In	46	1.31	1.63	12.2	7.6	7.5	4.1	8.2	4.7
Au-4	Au-12.3Cu-2.23In	47	1.22	1.73	12.0	8.1	6.5	3.1	9.3	9.9
Au-4	Au-12.3Cu-2.23In	48	1.39	1.51	12.6	11.7	8.2	6.6	7.0	9.3
Au-5	Au-1.0Co	49	1.93	1.91	10.3	9.7	7.9	4.1	8.2	11.7
Au-5	Au-1.0Co	50	2.05	2.20	9.6	6.7	8.1	3.5	7.7	8.8
Au-5	Au-1.0Co	51	1.93	2.29	10.6	6.5	9.5	4.0	8.3	7.1
Au-5	Au-1.0Co	52	1.80	2.18	10.4	10.0	6.8	4.1	7.0	8.2
Au-6	Au-1.0Co-1.0In	53	1.69	2.00	10.3	12.9	9.0	6.2	9.3	7.5
Au-6	Au-1.0Co-1.0In	54	1.78	2.02	9.2	13.5	10.1	7.1	8.2	9.3
Au-6	Au-1.0Co-1.0In	55	1.66	1.96	11.1	13.4	8.9	6.6	9.3	8.2
Au-6	Au-1.0Co-1.0In	56	1.87	1.99	7.9	5.3	10.1	7.3	7.0	7.2
EPI-100	Au-0.1Co/Cu	76	0.92	1.34	1.6	1.3	1.9	0.9	4.6	5.8
EPI-100	Au-0.1Co/Cu	80	1.03	1.44	1.1	2.3	2.1	1.2	4.5	6.3
EPI-200	Au-0.1Co/Cu	82	1.04	1.30	2.2	1.3	4.2	1.2	6.8	6.8
EPI-200	Au-0.1Co/Cu	84	1.15	1.41	1.7	1.3	4.3	2.0	5.2	6.4
EPI-50	Au-0.1Co/Cu	86	0.98	1.17	1.0	0.5	2.5	1.1	4.6	6.9
EPI-50	Au-0.1Co/Cu	88	0.96	1.15	0.9	0.6	2.5	2.8	4.6	8.1

TABLE XIII

(Continued)

Material		Test No.	Coefficient of Friction, μ_k		Static Contact Resistance, mΩ				Sliding Contact Noise, Peak to Peak, mΩ	
Designation	Nominal Composition		μ_1	μ_{100}	$(R_1)_{med.}$	ΔR	$(R_{100})_{med.}$	ΔR	ΔR_1	ΔR_{100}
EPII-100	Au-0.1Co/Ni	90	1.03	1.15	4.1	1.7	4.9	2.9	7.3	7.3
EPII-100	Au-0.1Co/Ni	92	1.04	1.17	3.2	1.7	6.0	2.0	7.8	7.2
EPII-200	Au-0.1Co/Ni	94	0.98	1.11	3.6	2.7	5.0	1.7	8.7	8.0
EPII-200	Au-0.1Co/Ni	96	0.95	1.28	3.5	2.7	5.1	3.7	8.1	5.8
EPII-50	Au-0.1Co/Ni	98	0.73	1.13	4.7	3.0	6.4	1.5	7.7	7.1
EPII-50	Au-0.1Co/Ni	100	0.72	1.11	4.5	3.1	7.2	1.7	8.0	7.3
EPIII-100	Au-1.0Co/Cu	102	0.96	1.72	1.7	0.9	2.0	2.0	4.6	5.8
EPIII-100	Au-1.0Co/Cu	104	0.84	1.68	2.1	1.3	1.6	1.0	4.6	4.6
EPIII-200	Au-1.0Co/Cu	106	1.02	0.68	2.8	1.8	4.4	4.5	5.3	5.8
EPIII-200	Au-1.0Co/Cu	108	1.08	1.81	2.6	2.2	1.8	2.0	6.0	6.0
EPIII-50	Au-1.0Co/Cu	110	0.83	1.19	1.3	0.4	2.0	2.6	4.6	6.9
EPIII-50	Au-1.0Co/Cu	112	0.76	1.23	1.9	0.2	4.7	3.4	5.2	6.9
Au-8	Au-0.5Zr	114	1.88	2.30	6.5	6.0	4.4	2.5	9.3	6.9
Au-8	Au-0.5Zr	116	1.63	2.50	8.0	5.7	4.9	2.8	9.3	7.5
Au-11	Au-2.5Zr	118	1.30	1.36	20.0	11.0	22.1	15.4	24.0	21.6
Au-11	Au-2.5Zr	120	1.25	1.42	24.0	13.2	20.7	16.0	24.0	24.2
Au-12	Au-19.4Ag-1.64Zr	122	0.96	0.44	8.9	7.9	16.1	18.6	10.6	42.5

For example the lightly alloyed materials such as Au-1.0Co, Au-0.5Zr, and Au-2.5Zr might be expected to have the least amount of residual oxide or other inorganic surface films and therefore relatively high friction. However, increased alloying levels of these reactive elements should favor residual or sliding-induced films. This may explain the much lower friction of the Au-2.5Zr alloy compared to Au-0.5Zr.

A similar argument may be used to explain the lower friction of Au-Ag-In versus Au-Ag. In N_2-20 O_2, Ag_2O is not thermodynamically stable at least as a thick film on these alloys. Alloying additions particularly those such as indium, having a low surface energy would be expected to increase surface reactivity significantly. In at least two cases--Au-Ag-In and Au-Co-In--such an effect was oberved.

The lower friction of the Au-12.1Cu alloy is believed to be due to a residual Cu_2O thin film (10-15A) and/or a sliding induced oxide film. The slightly higher friction of the Au-Cu-In alloy was thought to be due to an inhibition of Cu_2O formation. Such an effect was partially confirmed for "thick" oxide films in subsequent 200°C oxidation exposures.

As a second class of materials, the gold electroplates showed generally lower friction levels than the wrought alloys in the same environment. Initial values of μ_k were generally less than 1.1. The 100 cycle values were also significantly lower than comparable values for the wrought alloys. If the preceding conclusions about the friction of wrought alloys are correct, the results for the electroplates would suggest that these materials possess a unique surface chemistry which distinguishes the electroplates from the wrought alloys. Although the friction of the electroplates was high compared to what might be expected for boundary lubricated conditions ($\mu_k < 0.3$), the full significance of these results was realized in the wear and surface studies discussed below.

There appeared to be effects of both plating thickness and substrate material on friction. Friction generally tended to increase with increasing plating thickness and to decrease with increasing substrate hardness and/or elastic modulus. Lowest friction levels were obtained with the harder nickel (H_K = 270) substrate and the thinner electroplates. This is believed to be due to a decrease in the true contact area which should be determined in part by the mechanical properties of the substrate. With increasing plating thickness the substrate is expected to have a decreasing effect on the contact area with the result that friction and wear should more largely be dependent on the properties of the electroplate. The similarity in mechanical properties of the Au-Co electroplates and copper may explain the smaller effect of thickness than observed with the Au-Co/Ni systems.

All of the gold electroplates tended to show lower and more stable contact resistance than the wrought alloys. Noise levels were also very low compared to the design requirements of 10-20 mΩ which are presently being used in high reliability sliding systems. Most of the wrought alloys, however, would be acceptable on this basis.

The absolute value of contact resistance may be determined by two components--the constriction resistance and surface film contributions. The constriction resistance is directly proportional to the bulk resistivity of the material and inversely proportional to the size of the true contact area(s). It is, therefore, easy to understand that the magnitude of the static resistance should be different for alloys of different resistivities as shown in Table VI. This explains the high contact resistance of the Au-2.5Zr alloy which is close to the theoretical constriction resistance value of 20-30 mΩ. Of the wrought alloys, Au-19.0Ag which had the lowest resistivity also showed the lowest contact resistance. All of the other alloys showed about the expected values indicating very small surface film contributions.

It is interesting to note that the Au-Co electroplates gave contact resistances considerably lower than expected based on compositions. Median values for the Au-1.0Co electroplate (actually 0.76Co) were 1/4-1/5 those for the Au-1.0Co wrought alloy. This suggests that the resistivity of the Au-Co electroplates is much lower than that characteristic of a solid solution alloy. This leads to the conclusion that the electroplate may be structurally similar to a two-phase or overaged alloy containing virtually pure cobalt dispersed in a matrix of very high gold content.

It is also of interest to note the effect of the substrate on contact resistance. Electroplates on copper repeatedly showed lower contact resistance than those on nickel. This difference cannot be attributed to differences in the areas of contact and must be due primarily to the lower resistivity of copper. The explanation is believed to be in the fact that thin coatings do not satisfy the definition of a "long" constriction (Ref. 3) for which the simple constriction resistance equation applies. It is expected that in most practical plated systems (thickness < 200 microinches) the long constriction condition will not be met. Therefore, the substrate should affect contact resistance mainly by its bulk resistivity as well as by a small effect on the size of the contact areas.

Analysis of contact resistance stability is not quite as straightforward. It has been concluded that the major cause of variations in contact resistance in these sliding systems was a wear particle effect. The process of formation of loose wear debris and its finite residence time in the contact area is believed to have produced the observed variations. In extreme circumstances, it has been shown that very fine metallic wear particles can increase contact resistance and noise levels by nearly an order of magnitude above theoretical values (Ref. 4). This is due to the fact that each particle gives rise to a double constriction resistance proportional to the resistivity of the wear particle and inversely proportional to its mean size. An array of particles in the contact gap may increase the contact resistance far in excess of a single particle effect.

As a result of these effects the following resistance characteristics of sliding systems might be expected:

(1) Materials having a high bulk resisitivity may show increased noise levels and static resistance variations.

(2) A decrease in wear particle size should increase resistance variations.

(3) An increase in the number of loose wear particles should increase resistance variations.

(4) Alloying or surface chemistry changes tending to lower the surface energy of the system should decrease wear particle size (Ref. 5) and give a corresponding increase in resistance variations.

It is believed that most of these effects were actually observed in these studies. For example, the lowest noise levels in the wrought alloys were shown by the Au-19.0Ag alloy. Not only did this alloy have the lowest resistivity, it also gave (1) very few loose wear particles, and (2) the largest wear particles. Exactly the reverse situation was found for both the Au-2.5Zr and Au-19.4Ag-1.64Zr alloys.

All of the indium containing alloys showed increased values of ΔR which could be qualitatively related to a greater propensity for loose wear particle generation. This was believed related to (1) a decreased surface energy due to solid solution indium addition and/or (2) a decreased surface energy due to residual surface films.

The lowest values of ΔR were shown by the electroplates. This was attributed to (1) a low alloy resistivity, (2) a unique wear mode, and (3) a very small number of loose wear particles.

Friction and contact resistance in vacuum.--Results for the vacuum sliding experiments are given in Table XIV. In general the same conclusions were reached from these experiments that were reached in the N_2-20 O_2 tests.

For the wrought alloys except Au-19.0Ag both the initial friction levels and the rate of friction increase were greater in vacuum than in air. This is believed to reflect a greater rate of residual surface films disruption and a lower recontamination rate in vacuum.

Static contact resistance variations of the wrought alloys were lower in vacuum than in N_2-20 O_2. This was attributed to predominantly adhesive wear and very few wear particles. It is interesting to note that the indium-containing alloys continued to show higher values of ΔR suggesting a very definite effect of low surface energy additions on wear particle size.

Even in vacuum the electroplates performed quite well and were the only materials systems which gave initial friction values close to those obtained in N_2-20 O_2. The rate of friction increase was also comparable in both environments. This lends support to the thesis that the performance of these materials is due to an unique surface chemistry which is not related to the test environment but is a result of the electroplating process.

TABLE XIV

SUMMARY OF SIMILAR-METAL SLIDING CONTACT TEST DATA FOR VACUUM ENVIRONMENT

Material	Test No.	Vacuum, torr	Coefficient of Friction, μ_k			Static Contact Resistance, mΩ						Sliding Contact Noise Peak to Peak, mΩ		
			μ_1	μ_{100}	μ_{500}	$(R_1)_{med.}$	ΔR	$(R_{100})_{med.}$	ΔR	$(R_{500})_{med.}$	ΔR	ΔR_1	ΔR_{100}	ΔR_{500}
Au-1A	65	3.6×10^{-7}	1.93	2.20	2.77	5.2	5.3	2.6	1.6	1.3	3.5	9.3	9.2	8.2
Au-1A	66	1.8×10^{-7}	1.69	2.17	2.41	4.6	4.1	2.1	2.7	2.2	3.8	9.2	8.0	7.5
Au-2B	67	1.8×10^{-8}	1.48	2.10	3.20	7.0	6.7	4.6	1.6	1.3	4.9	8.9	11.2	8.9
Au-2B	68	3.8×10^{-8}	1.59	2.03	2.90	4.6	5.4	4.4	2.5	2.0	2.0	11.0	9.3	10.4
Au-3	69	9.0×10^{-8}	1.50	2.00	2.25	8.9	11.2	4.3	3.8	2.7	2.6	11.9	7.1	7.1
Au-3	70	3.6×10^{-8}	1.39	2.15	2.40	7.2	10.1	2.9	2.8	1.9	1.4	9.3	5.8	7.0
Au-4	71	4.0×10^{-8}	1.03	1.80	1.80	9.8	11.7	6.3	6.7	3.1	2.2	11.4	11.4	18.5
Au-4	72	1.0×10^{-8}	1.32	1.75	1.93	8.0	8.6	4.7	8.2	3.3	2.0	12.5	11.4	13.6
Au-5	73	1.0×10^{-7}	2.16	2.50	2.72	9.1	6.5	4.4	4.5	3.9	1.2	10.2	9.1	7.4
Au-5	74	3.6×10^{-7}	1.93	2.38	3.40	9.5	4.6	5.6	5.0	2.2	3.8	10.4	9.3	6.9
Au-6	77	2.2×10^{-8}	1.73	2.30	2.76	8.5	11.7	6.0	4.3	1.8	1.6	11.6	8.1	6.9
Au-6	78	4.4×10^{-7}	1.74	1.97	2.89	10.4	10.6	6.7	5.1	3.7	2.0	11.6	9.3	6.9
EPI-100	75	1.4×10^{-7}	1.17	1.17	1.11	1.0	1.2	2.7	2.8	5.3	2.4	4.5	6.8	6.8
EPI-100	79	5.8×10^{-8}	1.45	1.45	1.59	1.3	0.9	2.2	1.5	3.5	1.5	5.2	5.8	6.9
EPI-200	81	7.6×10^{-8}	1.37	1.63	1.89	1.6	1.6	2.3	2.7	1.6	1.6	5.7	6.8	6.8
EPI-200	83	8.6×10^{-8}	1.63	1.63	1.83	2.0	1.5	2.2	3.7	1.8	1.1	4.5	6.3	6.3
EPI-50	85	1.0×10^{-7}	1.37	1.17	1.44	0.9	0.4	2.1	4.6	2.0	4.2	4.6	9.3	9.8
EPI-50	87	7.2×10^{-8}	1.22	1.28	2.05	0.9	1.8	1.8	2.9	1.1	1.4	4.6	8.7	5.8
EPII-100	89	5.7×10^{-8}	1.15	1.35	1.48	3.9	2.4	5.8	1.9	5.3	2.1	6.9	6.9	8.1
EPII-100	91	6.0×10^{-8}	1.19	1.20	1.33	3.2	3.8	6.8	2.7	5.1	1.9	7.1	7.1	8.3
EPII-200	93	9.0×10^{-8}	1.13	1.26	1.32	4.0	2.9	4.4	2.9	4.1	2.5	7.2	7.2	8.4
EPII-200	95	1.0×10^{-7}	0.93	1.19	1.19	6.9	3.2	5.4	3.0	4.7	2.8	8.3	7.1	7.1
EPII-50	97	2.0×10^{-7}	0.95	1.28	1.35	3.7	3.7	6.3	2.6	5.0	4.0	6.9	6.4	7.5
EPII-50	99	8.0×10^{-8}	0.87	1.12	1.30	5.7	3.7	5.8	2.9	5.1	3.0	8.3	7.1	7.1
EPIII-100	101	2.8×10^{-7}	1.24	1.72	1.37	2.1	0.7	2.1	1.3	6.0	3.3	4.6	6.4	6.9
EPIII-100	103	2.0×10^{-7}	1.06	1.57	1.73	2.4	1.5	2.7	1.5	5.7	1.4	4.6	5.8	5.8
EPIII-200	105	4.6×10^{-7}	1.27	1.65	2.04	2.4	1.3	2.8	2.8	1.9	1.9	5.2	5.8	5.8
EPIII-200	107	5.0×10^{-8}	1.13	1.59	1.70	2.9	1.2	3.2	1.9	3.6	3.4	5.8	7.0	7.0
EPIII-50	109	1.0×10^{-7}	1.00	1.53	1.77	1.6	0.7	1.6	0.7	2.8	2.9	4.6	4.6	6.9
EPIII-50	111	1.0×10^{-7}	0.95	1.23	2.24	1.4	0.9	4.9	4.6	1.6	4.7	5.7	8.9	7.7
Au-8	113	7.2×10^{-8}	1.83	2.34	2.24	6.7	9.0	3.3	6.0	3.6	3.4	9.3	6.9	6.9
Au-8	115	1.0×10^{-8}	1.88	2.40	2.40	6.6	7.5	2.8	2.8	3.2	2.0	8.1	5.2	7.1
Au-11	117	2.0×10^{-7}	1.23	1.62	1.79	24.0	16.5	18.5	12.3	17.8	12.4	16.8	19.2	19 2
Au-11	119	1.0×10^{-7}	1.30	1.47	2.17	22.3	13.8	20.6	11.8	17.8	14.2	21.6	20.4	19.2
Au-12	121	2.6×10^{-7}	1.04	1.28	2.09	8.2	2.7	7.9	3.0	7.9	6.1	9.0	10.4	9.8

Wear.--Results of wear scar measurements are given in Tables XV and XVI. In both environments the data can clearly be separated into two groups--one for the wrought alloys and another for the electroplates. While differences were found within each group, as expected, the electroplates consistently gave much lower wear rates.

No correlation of wear rates with either friction or hardness was apparent. While data for the wrought alloys show the general trend toward lower wear with increasing hardness, exceptions were observed. For example, the wear rate of the hardest alloy--Au-Cu-In--was higher than the Au-Cu binary alloy. The Au-Co-In alloy which had a relatively low hardness showed one of the lower wear rates while the binary Au-Co alloy gave one of the highest rates.

Such "reversals" in wear characteristics from classical behavior have been commonly observed among specific classes of contact alloys such as the gold-base alloys as one class and palladium-base alloys as another. While hardness is of unquestionable importance to contact wear, surface chemistry and surface mechanical properties are believed to be of even greater importance in determining contact wear characteristics.

In vacuum the differences in wear rates among the wrought alloys were considerably less than in N_2-20 O_2. This was probably due to a lower recontamination rate by residual gases resulting in a smaller influence of surface films.

Photographs of typical wear features on each of the wrought alloys are shown in Figures 3 and 4. The severe wear and relatively large amounts of loose wear debris particularly in N_2-20 O_2 are evident.

In contrast is the less severe wear of all Au-Co electroplates as shown in Figures 5-7. It should be noted that these photographs are at higher magnification than used for the wrought alloys. The wear features of the electroplates assumed an almost burnished appearance somewhat characteristic of gold lubricated by a marginal boundary lubricant. This is shown in Figures 5b-e for the 100 and 200-microinch plates.

Within 100 cycles of sliding in N_2-20 O_2 breakthrough to the substrate occurred on the 50-microinch Au-0.1Co/Cu plate resulting in the severe wear shown in Figure 5a. The dark color of the wear track was produced by exposing the specimen to flowers of sulfur in order to demonstrate the consequence of severe wear. Exposures of the thicker plates failed to produce visible tarnish films.

For vacuum sliding the wear mode was unchanged at least up to the point of coating breakthrough. The 50-microinch coating (not shown) gave catastrophic failure, and the 100-microinch plate showed definite breakthrough within 500 cycles (Figure 6d). Relatively good durability in vacuum sliding was shown by the 200-microinch plate.

The lower wear rates for Au-0.1Co on nickel are evident in Figure 7 particularly for the thinner plates. Catastrophic failure did not occur in either N_2-20 O_2 or vacuum with the harder nickel substrate, at all thicknesses.

TABLE XV

SUMMARY OF SIMILAR-METAL WEAR DATA FOR N_2-20 O_2 ENVIRONMENT

Material		Test No.	Wear Scar Width After 100 Cycles (cm)	Initial Microhardness, $(H_k)_{100}$
Designation	Nominal Composition			
Au-1A	Au-19.0Ag	37	0.0145	110
Au-1A	Au-19.0Ag	38	0.0143	110
Au-2B	Au-12.1Cu	39	0.0089	225
Au-2B	Au-12.1Cu	40	0.0082	225
Au-3	Au-19.3Ag-2.05In	41	0.0127	157
Au-3	Au-19.3Ag-2.05In	42	0.0115	157
Au-4	Au-12.3Cu-2.23In	43	0.0107	235
Au-4	Au-12.3Cu-2.23In	44	0.0112	235
Au-3	Au-19.3Ag-2.05In	45	0.0135	157
Au-3	Au-19.3Ag-2.05In	46	0.0127	157
Au-4	Au-12.3Cu-2.23In	47	0.0107	235
Au-4	Au-12.3Cu-2.23In	48	0.0110	235
Au-5	Au-1.0Co	49	0.0186	102
Au-5	Au-1.0Co	50	0.0184	102
Au-5	Au-1.0Co	51	0.0157	102
Au-5	Au-1.0Co	52	0.0160	102
Au-6	Au-1.0Co-1.0In	53	0.0089	131
Au-6	Au-1.0Co-1.0In	54	0.0094	131
Au-6	Au-1.0Co-1.0In	55	0.0097	131
Au-6	Au-1.0Co-1.0In	56	0.0099	131
EPI-100	Au-0.1Co/Cu	76	0.0057	150[a]
EPI-100	Ditto	80	0.0055	150
EPI-200	"	82	0.0060	150
EPI-200	"	84	0.0051	150
EPI-50	"	86	0.0094	150
EPI-50	"	88	0.0096	150
EPII-100	Au-0.1Co/Ni	90	0.0056	150
EPII-100	Ditto	92	0.0071	150
EPII-200	"	94	0.0053	150
EPII-200	"	96	0.0046	150
EPII-50	"	98	0.0056	150
EPII-50	"	100	0.0058	150
EPIII-100	Au-1.0Co/Cu	102	0.0063	195
EPIII-100	Ditto	104	0.0061	195

(a) Estimated value corrected to 100-gram Knoop.

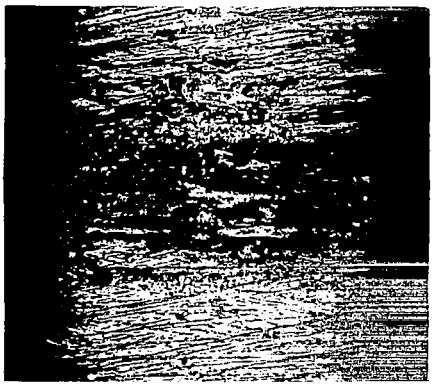
TABLE XVI

SUMMARY OF SIMILAR-METAL WEAR DATA FOR VACUUM ENVIRONMENT

Material		Test	Vacuum,	Wear Scar Width
Designation	Nominal Composition	No.	torr	After 500 Cycles, cm
Au-1A	Au-19.0Ag	65	3.6×10^{-7}	0.0371
Au-1A		66	1.8×10^{-7}	0.0381
Au-2B	Au-12.6Cu	67	2.0×10^{-8}	0.0328
Au-2B		68	3.8×10^{-8}	0.0225
Au-3	Au-19.3Ag-2.05In	69	9.0×10^{-8}	0.0280
Au-3		70	3.6×10^{-8}	0.0292
Au-4	Au-12.3Cu-2.23In	71	4.0×10^{-8}	0.0201
Au-4		72	1.0×10^{-8}	0.0268
Au-5	Au-1.0Co	73	1.0×10^{-7}	0.0440
Au-5		74	3.6×10^{-7}	0.0260
Au-6	Au-1.0Co-1.0In	77	2.2×10^{-8}	0.0226
Au-6		78	4.4×10^{-7}	0.0254
EPI-100	Au-0.1Co/Cu	75	1.4×10^{-7}	0.0120
EPI-100	Ditto	79	5.8×10^{-8}	0.0117
EPI-200	"	81	7.6×10^{-8}	0.0085
EPI-200	"	83	8.6×10^{-8}	0.0084
EPI-50	"	85	1.0×10^{-7}	0.0228
EPI-50	"	87	7.2×10^{-8}	0.0239
EPII-100	Au-0.1Co/Ni	89	5.7×10^{-8}	0.0132
EPII-100	Ditto	91	6.0×10^{-8}	0.0130
EPII-200	"	93	9.0×10^{-8}	0.0101
EPII-200	"	95	1.0×10^{-7}	0.0097
EPII-50	"	97	2.0×10^{-7}	0.0157
EPII-50	"	99	8.0×10^{-8}	0.0113
EPIII-100	Au-1.0Co/Ni	101	2.8×10^{-7}	0.0078
EPIII-100	Ditto	103		0.0082

Moveable

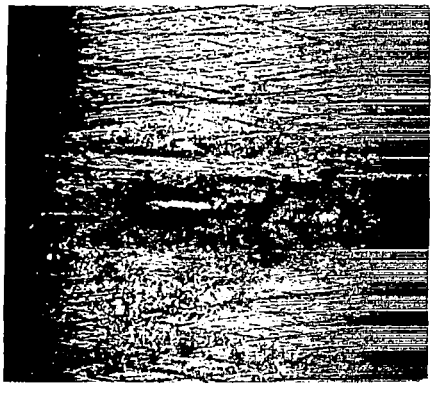
Rider



100X

a. Au-19.0Ag

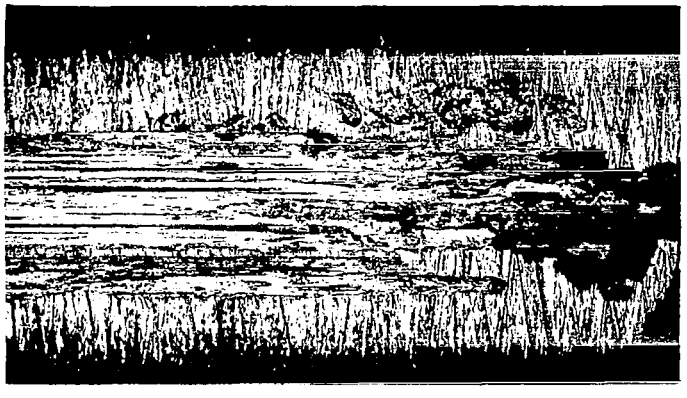
100X

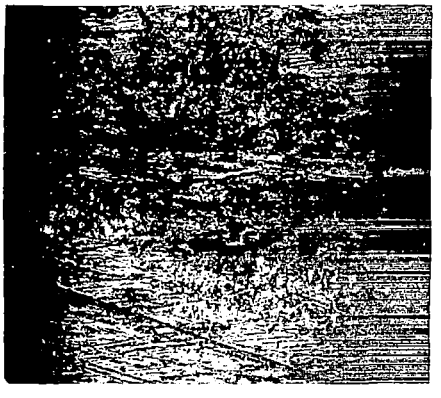


100X

b. Au-19.3Ag-2.05In

100X





100X

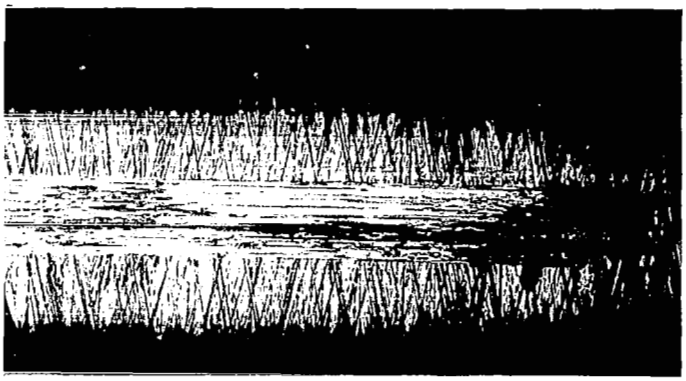
c. Au-1.0Co

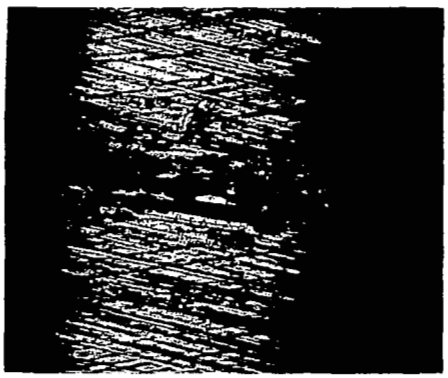
100X

FIGURE 3. WEAR OF WROUGHT GOLD ALLOYS IN N_2-20 O_2, 100 CYCLES

Moveable

Rider

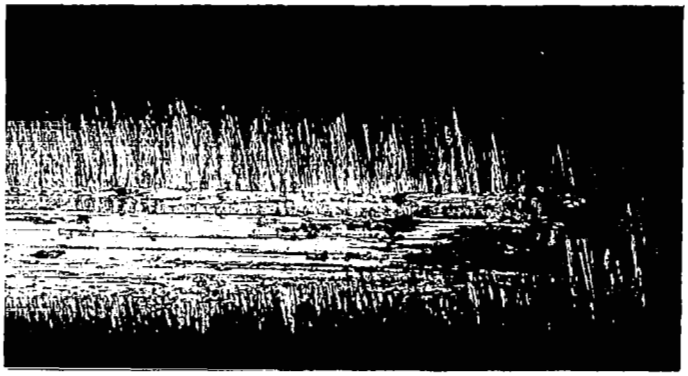


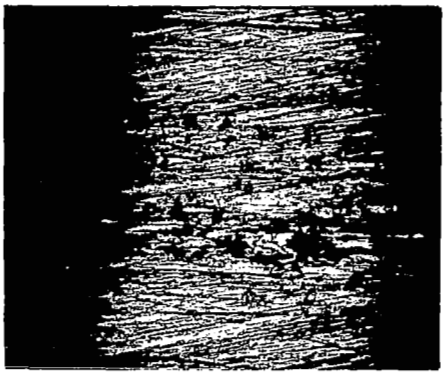


100X

100X

d. Au-12.1Cu





100X

100X

e. Au-12.3Cu-2.23In

200X

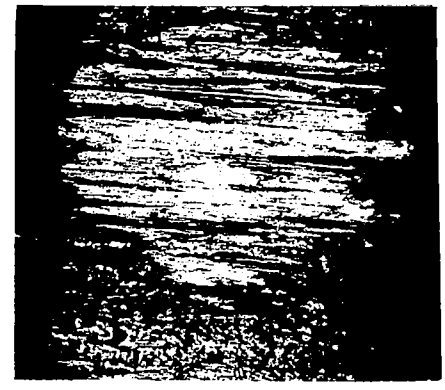
200X

f. Au-1.0Co-1.0In

FIGURE 3. (CONTINUED)

Moveable

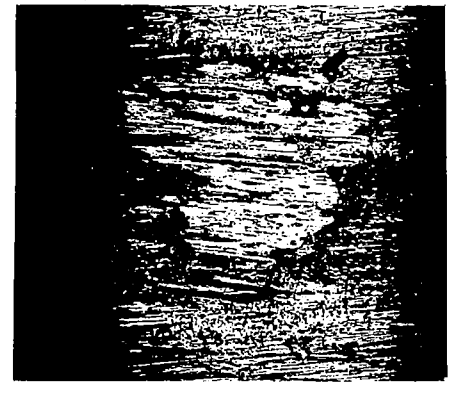
Rider



100X

100X

a. Au-19.0Ag



100X

100X

b. Au-19.3Ag-2.05In

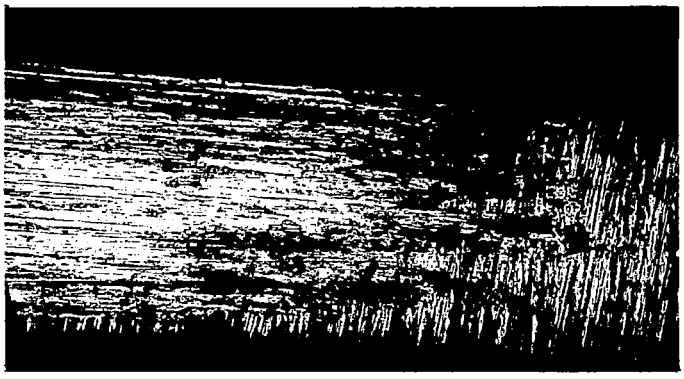
100X

100X

c. Au-1.0Co

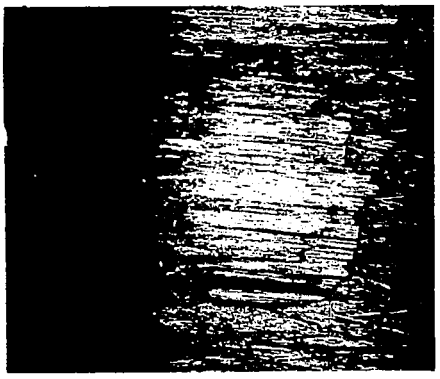
FIGURE 4. WEAR OF WROUGHT GOLD ALLOYS IN VACUUM, 500 CYCLES

Moveable



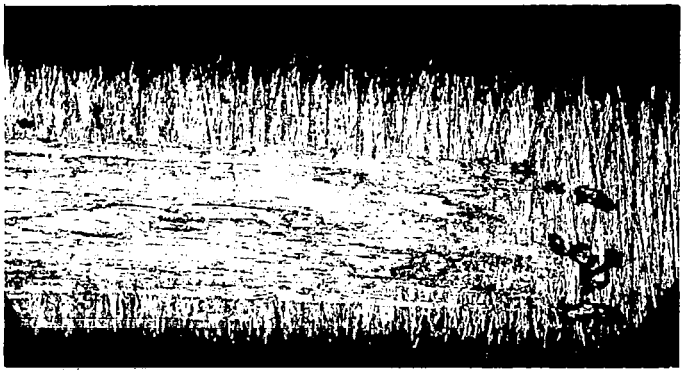
100X

Rider

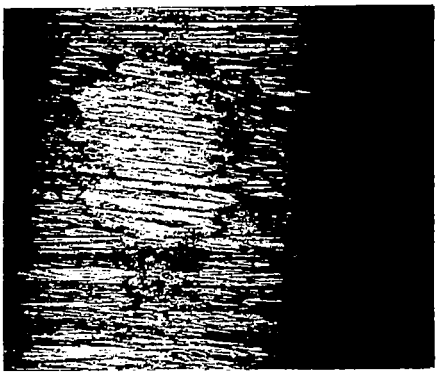


100X

d. Au-12.1Cu



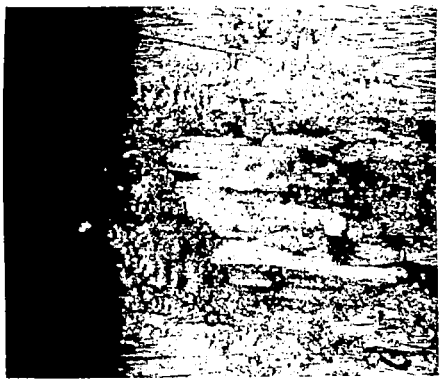
100X



100X

e. Au-12.3Cu-2.23In

100X



100X

f. Au-1.0Co-1.0In

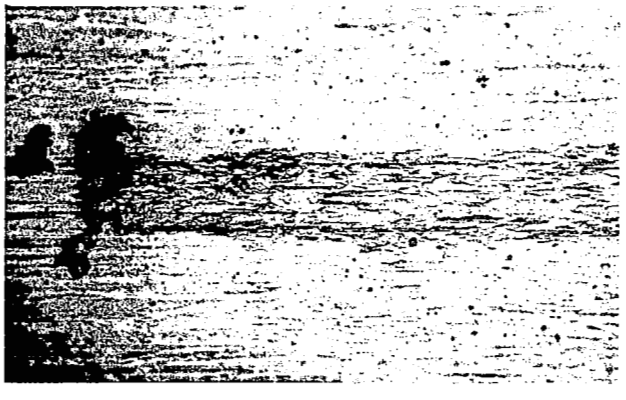
FIGURE 4. (CONTINUED)

End of Wear Track

Middle of Wear Track

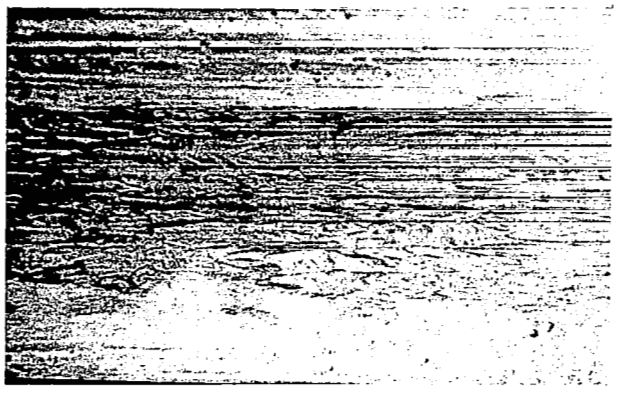
200X

a. 50 Microinches



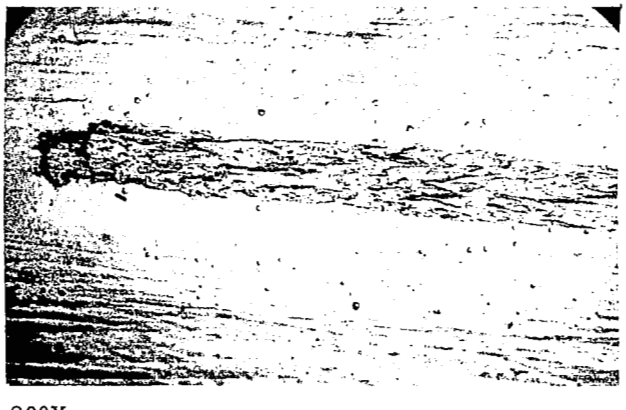
200X

b. 100 Microinches



400X

c. 100 Microinches



200X

d. 200 Microinches

400X

e. 200 Microinches

FIGURE 5. WEAR OF Au-0.1Co/Cu ELECTROPLATES IN N_2-20 O_2, 100 CYCLES

End of Wear Track

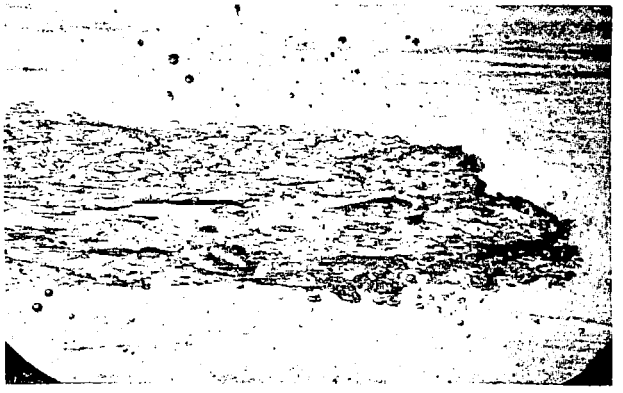
200X

a. 100 Microinches

Middle of Wear Track

200X

b. 100 Microinches



200X

c. 200 Microinches

400X

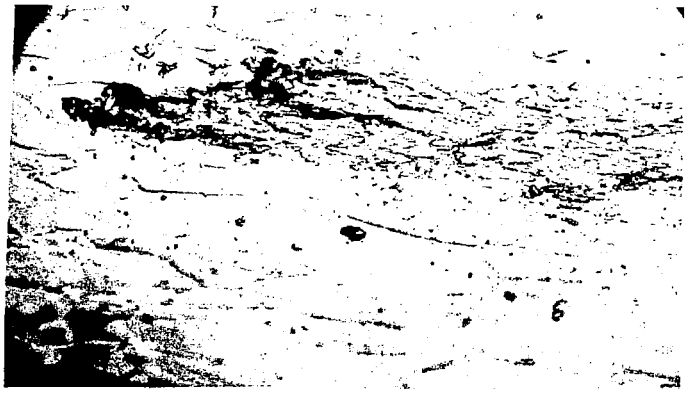
d. 200 Microinches

FIGURE 6. WEAR OF Au-0.1Co/Cu ELECTROPLATES IN VACUUM, 500 CYCLES

End of Wear Track

200X

a. 50 Microinches



200X

b. 100 Microinches

200X

c. 200 Microinches

FIGURE 7. WEAR OF Au-0.1Co/Ni ELECTROPLATES IN N_2-20 O_2, 100 CYCLES

The preceding microscopic observations were in good agreement with electrographic prints made of the wear tracks. In N_2-200_2 substrate breakthrough was not evident on the 100 or 200-microinch plates on copper or nickel. Excessive base metal was exposed with the 50-microinch plates on copper but with nickel the defect density was only slightly greater than the initial porosity level.

In vacuum the 200-microinch plate on copper gave isolated indications of breakthrough. The same nominal thickness on nickel appeared to remain defect-free after 500 cycles.

A complete analysis of the wear characteristics of Au-1.0Co was not made. Wear rates and degree of protection of the substrate appeared to be inferior to the Au-0.1Co alloy. For this and other reasons to be presented the Au-1.0Co alloy was not considered to be an acceptable material to meet the program requirements.

Dissimilar-Metal Contact Sliding

The results of the several dissimilar-metal tests are given in Tables XVII and XVIII. In all of these experiments the hard, complex alloys Au-7 and Pd-1 were used only as the stationary contact members. The reason for this was to study the effects of directional transfer from the movable member to the stationary member on friction and wear. In particular, it was of interest to determine the differences between (1) a totally similar metal sliding system, and (2) a "similar-metal" system produced by thin film directional trnasfer. In order to achieve the second type of system previous studies have indicated that the following conditions must be met:

(1) The material being transferred should be the contact member having the larger apparent contact area.

(2) The surface work hardening rate of the material being transferred should be much greater than that of the other contact member.

Both of these conditions are satisfied for the materials systems/geometry listed in Table XVII.

Examination of test specimens confirmed that directional transfer did occur in the manner expected. A uniform film of the movable contact material developed rapidly on the stationary member. Judging by the test data in Table XVII however, the results were nearly identical with earlier similar metal tests. Apparently the transfer film which formed was too thick to impart any degree of metallic thin film "lubrication".

Wear rates of the movable members were significantly lower in the dissimilar-metal tests. This, however, was the only advantage shown by these materials systems.

TABLE XVII

SUMMARY OF DISSIMILAR-METAL SLIDING CONTACT TEST DATA FOR N_2-20 O_2 ENVIRONMENT

Material		Test	Coefficient of Friction, μ_k		Static Contact Resistance, mΩ				Sliding Contact Noise Peak to Peak, mΩ	
Moveable	Stationary	No.	μ_1	μ_{100}	$(R_1)_{med}$	ΔR	$(R_{100})_{med}$	ΔR	ΔR_1	ΔR_{100}
Au-2B	Au-7	57	1.42	1.48	11.5	11.0	7.3	3.2	11.8	8.3
Au-2B	Au-7	58	1.26	1.64	11.7	8.7	8.5	3.9	10.6	9.4
Au-1A	Au-7	59	2.14	2.02	6.7	4.1	4.7	1.9	5.7	6.8
Au-1A	Au-7	60	1.83	2.08	7.0	3.7	3.5	2.4	6.8	6.3
Au-2B	Pd-1	61	1.12	1.26	11.3	6.5	7.4	5.1	11.8	8.8
Au-2B	Pd-1	62	1.19	1.88	8.0	5.6	6.4	3.0	14.2	9.4
Au-1A	Pd-1	63	1.63	2.00	8.5	2.9	4.9	3.0	8.5	7.3
Au-1A	Pd-1	64	1.94	2.07	5.6	5.2	5.1	1.9	9.7	8.5

TABLE XVIII

SUMMARY OF DISSIMILAR-METAL WEAR DATA
FOR N_2-20 O_2 ENVIRONMENT

Material		Test	Wear Scar Width
Stationary	Movable	No.	After 100 Cycles, cm
Au-7	Au-2B	57	0.0098
Au-7	Au-2B	58	0.0100
Au-7	Au-1A	59	0.0094
Au-7	Au-1A	60	0.0080
Pd-1	Au-2B	61	0.0092
Pd-1	Au-2B	62	0.0092
Pd-1	Au-1A	63	0.0147
Pd-1	Au-1A	64	0.0163

Environmental Studies

Selected wear test specimens which had been tested for 100 cycles in N_2-20 O_2 were subjected to two environmental exposures together with control specimens of the same alloy. The control specimens were not wear tested. These exposures consisted of (1) 120 hours in flowers of sulfur at 30°C and (2) 1000 hours in N_2-20 O_2 at 200°C. Contact resistance measurements were made along the wear tracks of the test specimens and on control specimens. Additional control specimens of those alloys containing copper and/or silver were analyzed by electrolytic reduction in order to determine amounts of reducible oxide and/or sulfide films.

As the research progressed it became clear that the major environmental problem was the 200°C oxidation resistance. Therefore, later environmental tests particularly on the electroplates concentrated on the oxidation characteristics of control specimens.

Flowers of sulfur.--The results of contact resistance measurements on specimens exposed to the flowers of sulfur environment are given in Tables XIX-XXI. None of the control specimens showed any measure of degradation as a result of this test. All maintained a range of contact resistance values nearly identical with values for clean conditions (not given).

Table XIX shows that the wear tracks of the wrought alloys generally showed both a greater variation in contact resistance and higher median values than shown by the control specimens. The data cannot be attributed directly to the highly worked condition of the wear track. Two explanations are possible. First the higher resistance values may be due to a significant wear debris contribution. The basis for this was given earlier.

The second possibility is an increased surface film contribution due to higher local rates of tarnishing in the wear track. It has been shown that for Au-Ag and Au-Cu alloys tarnish rates are very sensitive to the metallurgical condition of the material (Ref. 6). Any operation tending to increase the lattice defect density such as cold working can lead to a very substantial increase in the rate of surface film growth. This was actually observed on the Au-12.1Cu binary alloy. Control specimens tested for 120 hours developed a film consisting of $\sim$ 45 A Cu_2S and 11 A Cu_2O. At this thickness the film was not visible. Wear test specimens tested in a similar manner showed a clear delineation of the wear area by the development of a film(s) which were thick enough to be readily visible.

It is uncertain which factor accounted for the increased wear track resistance but the surface film contribution was probably dominant for the silver and copper-containing alloys. These data show that the reactivity of the wear track may be considerably different from the surrounding surface area. This is of great practical importance for it indicates that the true susceptibility of a material toward environmental contamination cannot be judged solely from measurements on control or as-manufactured surfaces. The final measure of the real merits of a contact material should include data on surfaces initially subjected to actual wear conditions.

TABLE XIX

CONTACT RESISTANCE OF PRECIOUS METAL ALLOYS
AFTER 120 HOURS IN N_2-20 O_2-40 ppb S_8 AT 30°C

Material			Contact Resistance[a] of					
			Control Specimen[b]			Wear Track of Test Specimen[c]		
Designation	Nominal Composition	Test No.	R_{max}	R_{min}	R_{med}	R_{max}	R_{min}	R_{med}
Au-1A	Au-19.0Ag	35	3.33	2.00	2.37	14.2	1.76	3.16
		36	3.02	2.05	2.46	10.7	2.14	3.09
Au-2B	Au-12.1Cu	39	2.80	2.60	2.74	5.35	3.31	5.28
		40	3.24	2.60	2.74	16.6	2.81	5.10
Au-3	Au-19.3Ag-2.05In	45	3.23	2.44	2.82	3.89	2.72	3.06
		46	3.61	2.88	3.44	6.10	3.06	3.61
Au-4	Au-12.3Cu-2.23In	47	4.00	3.33	3.68	4.34	3.47	4.00
		48	5.00	3.62	3.75	9.06	3.25	4.06
Au-5	Au-1.0Co	49	3.62	1.92	2.26	7.94	4.46	5.76
		50	4.62	3.76	4.15	8.48	4.17	7.92
Au-6	Au-1.0Co-1.0In	55	4.00	2.10	3.70	7.31	1.72	3.05
		56	7.12	2.10	2.25	5.76	1.80	2.12
Au-8	Au-0.5Zr	114	6.21	2.35	4.20	11.8	4.60	8.00
		116	5.85	3.20	4.15	4.80	3.12	4.16
Au-11	Au-2.5Zr	118	10.8	8.21	9.90	12.2	8.15	10.2
		120	10.0	8.91	9.00	15.4	9.51	13.5

(a) Measured against pure gold at 10 grams.

(b) Control specimen not wear tested.

(c) 100 cycles in N_2-20 O_2 at 760 mm Hg, 25°C.

TABLE XX

CONTACT RESISTANCE OF Au-0.1Co ELECTROPLATES AFTER 120 HOURS IN FLOWERS OF SULFUR AT 30°C

Plating Thickness, microinches	Substrate	Test No.	Contact Resistance of[a] Control Specimen[b] R_{max}	R_{min}	R_{med}	Wear Track Test Specimen[c] R_{max}	R_{min}	R_{med}
50	Cu	86	2.0	0.8	1.2	11.9	1.5	2.2
		88	2.7	0.9	1.2	16.9	0.9	7.7
100	Cu	76	2.1	1.0	1.4	1.8	1.0	1.7
		80	2.0	0.9	1.6	2.3	0.9	1.6
200	Cu	82	2.5	1.1	1.7	2.9	1.2	1.9
		84	2.4	1.0	1.6	2.6	1.4	1.8
50	Ni	98	7.7	1.9	3.5	15.9	5.3	6.3
		100	4.7	2.5	3.2	13.0	2.7	3.6
100	Ni	98	4.7	1.7	2.5	3.1	1.7	2.7
		92	4.3	0.9	1.6	11.4	2.3	3.5
200	Ni	94	4.5	2.1	3.2	12.5	2.8	2.6
		96	5.3	1.9	3.4	4.3	2.1	3.8

(a) Measured against pure gold at 10 grams.

(b) Control specimen not wear tested.

(c) 100 cycles in N_2-20 O_2 at 760 mm Hg, 25°C.

TABLE XXI

CONTACT RESISTANCE OF Au-1.0Co ELECTROPLATES ON COPPER AFTER 120 HOURS IN FLOWERS OF SULFUR AT 30°C

Plating Thickness, microinches	Test No.	Contact Resistance of[a] Control Specimen[b] R_{max}	R_{min}	R_{med}	Wear Track of Test Specimen[c] R_{max}	R_{min}	R_{med}
100	102	3.5	2.1	2.6	3.2	1.7	2.6
	104	2.6	1.7	2.5	3.8	2.1	2.5
200	106	3.5	2.0	2.7	3.1	2.0	2.5
	108	3.1	1.7	2.6	3.6	1.9	2.5
50	110	2.1	1.2	1.7	2.8	1.3	1.8
	112	2.5	1.5	2.2	2.9	1.0	2.0

(a) Measured against pure gold at 10 grams.

(b) Control specimen not wear tested.

(c) 100 cycles in N_2-20 O_2 at 760 mm Hg, 25°C.

The wear track reactivity of electroplated specimens, particularly for the thicker deposits, was generally less affected by the sulfur exposure than was observed for wrought specimens. Increased resistance in the wear track (as opposed to control specimens) was most prominent on the 50-microinch Au-0.1Co. This was attributed to breakthrough to and subsequent tarnishing on the base metal. It is believed that the measured values on the 50-microinch systems are lower than actual values that would more properly indicate the true merit of these systems. Because of the geometry of wear it was not possible to bring the gold probe down into the wear track. The actual areas of contact were the sharp, high points around the edges of the wear track. Therefore, the heavily tarnished areas were actually not measured. This situation did not arise with the thicker plates which gave uniform, mild wear, and no visible tarnish films.

The flowers of sulfur test failed to show any significant difference in reactivity of the two Au-Co electroplated alloys. Table XXI seems to suggest an improved performance of the Au-1.0Co at low plating thicknesses on the basis of contact resistance in the wear track. As with the Au-0.1Co at 50 microinches heavy base metal tarnishing was observed at areas of breakthrough. It is believed that these areas were not actually probed during contact resistance measuremnets due to the geometry of wear. Therefore, the actual resistance of a contact system of 50 microinches Au-1.0Co would probably have been much greater and unacceptable.

Oxidation.--A 200°C exposure in N_2-20 O_2 proved to be the most severe test of both the wrought alloys and the electroplates. Among the wrought alloys the Au-19.0Ag gave no resistance increase for either the control or wear test specimens as shown in Table XXII. This was expected since Ag_2O is thermodynamically unstable on this alloy at 200°C. Alloying additions of indium gave high contact resistance on this alloy. This was believed to be due to the formation of a thin (< 100 A) In_2O_3 film which could not be measured by electrolytic reduction.

The Au-12.1Cu alloy developed very high contact resistance attendant with the formation of about 1200-1500 A Cu_2O + CuO after 1000 hours. Indium additions to Au-Cu sharply decreased the rate of copper oxide(s) formation but still gave high contact resistance. This was attributed to the formation of In_2O_3 or a complex copper-indium oxide film.

The Au-1.0Co wrought alloy was an interesting material. After a 1000-hour exposure a visibly thick film had formed. However, no high resistance events were measured on either the control specimen or the wear area. This does not mean that the film (CoO) was conductive. Instead, force versus contact resistance measurements indicated that film breakdown occurred at forces less than 1 gram. Conduction was probably by metallic asperity contact. In this respect oxide films on the Au-Co alloy appear to be much like Ag_2S films on silver. Very thick films are required even under dry circuit conditions before significant increases in contact resistance occur. In both cases this can be attributed to the mechanical rather than electrical properties of the films.

These results although interesting do not automatically mean that Au-Co alloys are suitable for 200°C service. The fact that visible films do form

TABLE XXII

CONTACT RESISTANCE OF PRECIOUS METAL ALLOYS AFTER 1000-HOUR OXIDATION[a] AT 200°C

Material		Test No.	Contact Resistance of Control Specimen			Contact Resistance of Wear Track of Test Specimen[b]		
Designation	Nominal Composition		R_{max}	R_{min}	R_{med}	R_{max}	R_{min}	R_{med}
Au-1A	Au-19.0Ag	37	4.20	2.61	2.88	5.60	3.00	3.54
		38	3.60	2.88	3.32	5.10	2.52	3.66
Au-2B	Au-12.1Cu	33	2790.	42.6	2100.	165.	36.0	51.5
		34	1620.	12.1	271.	5830.	49.3	362.
Au-3	Au-19.3Ag-2.05In	41	97.6	6.50	22.0	114.	7.1	66.0
		42	20.9	4.10	7.8	143.	20.9	102.
Au-4	Au-12.3Cu-2.23In	43	375.	62.5	73.5	2375.	53.1	226.
		44	111.	11.3	19.7	5840.	23.8	717.
Au-5	Au-1.0Co	51	5.0	3.3	4.2	9.2	2.5	3.8
		52	2.9	2.3	2.7	6.8	2.7	4.2
Au-6	Au-1.0Co-1.0In	53	7.9	4.6	5.3	55.3	3.9	7.4
		54	18.8	6.7	8.3	96.0	5.0	10.0
Au-7[c]	--	--	194.	31.8	55.8	--	--	--
		--	100.	33.9	61.5	--	--	--
Au-8	Au-0.5Zr	--	4.5	3.1	3.8	--	--	--
		--	6.2	3.0	4.1	--	--	--
Au-11	Au-2.5Zr	--	8.4	6.3	7.6	--	--	--
		--	9.4	4.2	6.3	--	--	--
Au-12	Au-19.4Ag-1.64Zr	--	5.9	4.8	5.8	--	--	--
		--	6.0	4.7	5.7	--	--	--
Pd-1[d]	--	--	8.3	6.5	7.5	--	--	--
		--	9.0	7.3	8.6	--	--	--

(a) N_2-20 O_2, 760 mm Hg; contact resistance measured against gold at 10 grams.

(b) 100 cycles in N_2-20 O_2 at 760 mm Hg; 25°C.

(c) Surface film contained at least 330 A Cu_2O and 215 A CuO.

(d) Survace film contained about 110 A Cu_2O.

presents the possibility that on repeated sliding the films could be compacted into the wear track. The repeated process of oxidation and compaction could lead to eventual failure by a high resistance surface layer. Such a failure mechanism is well known for Ag_2S films on silver.

Indium additions to the Au-Co binary alloy significantly increased wear track reactivity. Contact resistances of the Au-Co-In control specimens, although much lower, were believed to show a moderate surface film component.

The zirconium-containing alloys were particularly interesting. Not only did their contact resistance remain low during 1000 hours at 200°C, but no visible films were detected. Force resistance-measurements failed to indicate any film breakdown process at forces as low as 100 milligrams. Corresponding tests on the wear specimens were not completed which proved to be unfortunate in view of the very promising results on the control specimens. Considering the very stable electrical and mechanical properties of these alloys at 200°C further tests should be made to define their oxidation resistance particularly for (1) gold hardened by solid solution addition of < 1% Zr, (2) gold precipitation hardened by 1-2% Zr in order to decrease bulk resistivity, and (3) Au-Ag alloys hardened by zirconium.

The differences in oxidation resistance between the Au-Zr and Au-Co alloys may be due to the nature of the second phase which forms in these alloys. In the case of Au-Co this phase is almost pure cobalt but in Au-Zr it has the composition Au_3Zr. It is conceivable that in the Au-Co system, precipitation of the second phase precedes the oxidation reaction in which case oxidation of the cobalt-rich phase might be dominant. Precipitation rates in the Au-Zr alloys appear to be very low at 200°C but even if the Au_3Zr phase formed its oxidation resistance should be far better than that of "pure" cobalt.

A summary of the surface film analyses obtained by electrolytic reduction is given in Table XXIII. Cobalt, indium and zirconium compounds could not be determined.

Results of oxidation tests on the electroplates are given in Tables XXIV-XXVII. Additional data points at 90-120 hours were obtained for these systems in order to obtain a better view of the degradation kinetics.

None of the electroplates on copper was found to be acceptable for even short time (100 hour) service at 200°C. Data in Tables XXIV and XXVI show high contact resistance on both the Au-0.1Co and Au-1.0Co alloys after 113 and 90 hours, respectively. All specimens were uniformly discolored indicating the presence of a surface film. This was confirmed by electrolytic reduction which showed a considerable amount of copper oxides even on the 200-microinch plates.

Contact resistance values of the Au-1.0Co plate were much higher than for corresponding exposures of Au-0.1Co even for short exposures. This is believed to indicate an inherently poor oxidation resistance of the Au-Co electroplates at the 1.0 percent cobalt level. Partial confirmation was found in experiments with thick electroplates on nickel substrates. Contact resistance of a 200-microinch Au-0.1Co plate remained stable throughout 1000 hours of testing. One isolated measurement of 18.5 mΩ was made after 1000 hours (Table XXV) which

TABLE XXIII

ANALYSIS OF SURFACE FILMS AFTER 1000-HOUR OXIDATION AND 120-HOUR TARNISH TESTING[a]

Material		Film Thickness (A)/Composition					
		120-Hr Tarnish Test			1000-Hr Oxidation		
Designation	Composition, weight percent	Ag_2S	Cu_2S	Cu_2O	Ag_2O	Cu_2O	CuO
Au-1A	Au-19.0Ag	53	--	--	0	--	--
		50	--	--	0	--	--
Au-2B	Au-12.1Cu	--	44	11	--	1050	432
		--	46	11	--	980	360
Au-3	Au-19.3Ag-2.05In	34	--	--	0	--	--
		38	--	--	0	--	--
Au-4	Au-12.3Cu-2.23In	--	33	23[b]	--	79	216[c]
		--	38	28	--	80	206
Au-5	Au-1.0Co	--	--	--[d]			
		--	--	--	Visible oxide film could not be electrolytically reduced		
Au-6	Au-1.0Co-1.0In	--	--	--[d]			
		--	--	--[d]			

(a) Tests conducted on specimens not previously subjected to wear tests.

(b) Third film detected but not identified; estimated thickness 10-15 A.

(c) Third film detected but not identified; estimated thickness 40-50 A.

(d) No films detected by electrolytic reduction.

TABLE XXIV

OXIDATION OF Au-0.1Co ELECTROPLATES ON COPPER IN N_2-20 O_2 AT 200°C

Plating Thickness, microinches	Oxidation Time, hr	Contact Resistance, milliohms			Film Thickness, A	
		R_{max}	R_{min}	R_{med}	CuO	Cu_2O
50	113	6.7	4.4	5.0	18.	92.
	113	6.7	3.9	4.7	18.	93.
100	113	13.9	6.7	8.3	12.	46.
	113	38.8	13.3	19.3	14.	52.
200	113	73.9	8.3	43.2	0	80.
	113	153.	16.1	25.6	0	52.
50	1000	915.	13.6	21.2	297.	113.
	1000	∞	22.6	56.5	200.	151.
100	1000	45.9	10.2	20.0	158.	151.
	1000	20.4	3.0	8.6	187.	132.
200	1000	61.4	7.5	12.5	165.	53.
	1000	100.	9.4	60.8	70.	46.

(a) Measured against gold at 10 grams.

TABLE XXV

OXIDATION OF Au-0.1Co ELECTROPLATES ON NICKEL IN N_2-20 O_2 AT 200°C

Plating Thickness, microinches	Oxidation Time, hr	Contact Resistance, mΩ		
		R_{max}	R_{min}	R_{med}
50	120	69.0	5.7	25.6
	120	120.	9.8	34.2
100	120	79.6	6.1	9.7
	120	18.	8.9	15.6
200	120	6.8	3.2	4.8
	120	5.9	3.9	5.8
50	1000	3940.	2.4	3.9
	1000	4310.	2.5	4.2
100	1024	1545.	32.1	110.
	1024	42.4	5.8	16.7
	1024	129.	19.2	38.1
	1024	515.	3.0	21.8
200	1000	18.5[a]	2.6	5.3
	1000	4.4	1.6	3.2

(a) Single measurement > 7 mΩ.

TABLE XXVI

OXIDATION OF Au-1.0Co ELECTROPLATES ON COPPER IN N_2-20 O_2 AT 200°C

Plating Thickness, microinches	Oxidation Time, hr	Contact Resistance, mΩ			Film Thickness, A
		R_{max}	R_{min}	R_{med}	Cu_2O
50	90	6740.	41.6	69.6	60.
	90	∞	204.	2600.	65.
100	90	1080.	6.7	209.	52.
	90	84.7	6.1	33.6	50.
200	90	100.	16.3	24.2	25.
	90	79.6	10.6	23.8	35.
50	1000	4550.	135.	1450.	129.
	1000	∞	175.	209.	113.
100	1000	1810.	54.8	70.0	89.
	1000	2780.	216.	830.	94.
200	1000	3300.	12.1	78.6	76.
	1000	8400.	18.0	735.	86.

TABLE XXVII

OXIDATION OF Au-1.0Co ELECTROPLATES ON NICKEL IN N_2-20 O_2 AT 200°C

Plating Thickness, microinches	Oxidation Time, hr	Contact Resistance, mΩ		
		R_{max}	R_{min}	R_{med}
50	90	198.	12.2	22.6
	90	60.9	7.0	30.0
100	90	87.0	11.3	24.6
	90	63.5	14.1	18.9
200	90	8300.	58.0	59.5
	90	127.	23.6	48.7
50	1000	427.	65.2	78.2
	1000	510.	70.5	80.2
100	1000	∞	131.	4150.
	1000	9440.	16.2	291.
200	1000	∞	31.8	51.7
	1000	214.	17.3	21.4

could have been due to other forms of contamination such as particulates. Assuming that diffusion rates are similar in both gold alloys (which is supported by data for copper substrates) similar resistance levels might be expected from a substrate effect. High resistance of the 200-microinch Au-1.0Co/Ni after even 90 hours indicates alloy oxidation.

Plates < 200 microinches of Au-0.1Co gave high contact resistance although none showed visible films. It is known, however, that very thin films (< 30-40 A) of NiO will produce very high resistance in dry circuits due to their mechanical properties. Films of Cu_2O of similar thickness may give a barely perceptible increase in resistance.

It appears that the problems of elevated temperature alloy oxidation can be circumvented by the use of the Au-0.1Co alloy. This material also gave the best dry sliding characteristics of all the materials tested. However, for high reliability in elevated temperature applications the choice of a substrate material is critical. Nickel has long been used as a barrier material or substrate to minimize diffusion during processing and/or service. For applications at 200°C the choice of nickel must be questioned. The data indicate that thin plates (< 100-150 microinches) would be totally unacceptable. A minimum thickness of 200-250 microinches appears necessary for 1000-hour storage life at 200°C. However, this value does not consider the possibility of wear prior to elevated temperature exposure which would decrease the plating thickness in the contact area. Thicker plates which would be required to compensate for initial wear would generally be incompatible with present design practices and manufacturing technology.

CONCLUSIONS

As a result of these studies only one materials system has been defined which will meet most of the program requirements of:

(1) Low wear rates in both air and vacuum

(2) Low contact resistance and good contact resistance stability

(3) Resistance to environmental contamination

(4) Stability during 1000-hour exposures in air at temperatures up to 200°C.

The system of a Au-0.1Co alloy electroplate over nickel appears to meet the first three requirements for sliding durations to at least 100 and probably > 500 cycles in air and 500 cycles in high vacuum.

The fourth requirement cannot be met by this system for plating thicknesses < 100 microinches. The Au-0.1Co alloy appears to exhibit satisfactory oxidation resistance. Diffusion of substrate material via a "short-circuit"; i.e., grain boundary or dislocation, transport mechanism and subsequent

oxidation at the alloy surface has been identified as a major reliability problem. Plating thicknesses of 200-250 microinches would be the minimum recommended for 1000-hour storage life at 200°C. This assumes no contact wear prior to high temperature storage.

The Au-0.1Co system could not be recommended for high reliability applications in which the contacts would be:

(1) Operated either intentionally or unintentionally (due to vibration) at 200°C in air

(2) Operated at low temperatures and stored at 200°C for future use.

This system could be recommended for continuous use only at relatively low to moderate temperatures.

No materials system has, therefore, been defined which will meet all four requirements. The major problem remaining to be solved is that of high temperature substrate diffusion. Future efforts should be directed toward the definition of a diffusion barrier material suitable for 200°C service. Preliminary experiments have indicated that rhodium may be such a material.

Finally it should be noted that one important series of tests has not been made on the Au-0.1Co electroplates. This is a study of friction and wear after a 200°C exposure in air. While metallurgical changes at 200°C could affect performance another change could be of even greater importance. The sliding characteristics of this electroplate were attributed to an organic compound codeposited with the alloy. Nothing is known about the effects of high temperature on this organic or whether it would provide some degree of lubrication after air oxidation at 200°C. Further studies are needed to clarify this point before the Au-0.1Co could be qualified for elevated temperature service.

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